A Summary of Current Program, 7/1/63

and Preliminary Report of Progress

for 7/1/62 to 6/30/63

SOUTHERN UTILIZATION RESEARCH AND

DEVELOPMENT DIVISION

of the

AGRICULTURAL RESEARCH SERVICE

UNITED STATES DEPARTMENT OF AGRICULTURE

This progress report of U.S.D.A. and cooperative research is primarily a tool for use of scientists and administrators in program coordination, development and evaluation; and for use of advisory committees in program review and development of recommendations for future research programs.

The summaries of progress on U.S.D.A. and cooperative research include some tentative results that have not been tested sufficiently to justify general release. Such findings, when adequately confirmed will be released promptly through established channels. Because of this, the report is not intended for publication and should not be referred to in literature citations. Copies are distributed only to members of Department staff, advisory committee members and others having a special interest in the development of public agricultural research programs.

This report also includes a list of publications reporting results of U.S.D.A. and cooperative research issued between July 1, 1962, and June 30, 1963. Current agricultural research findings are also published in the monthly U.S.D.A. publication, Agricultural Research. This progress report was compiled in the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, New Orleans 70124, Louisiana.

UNITED STATES DEPARTMENT OF AGRICULTURE
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INTRODUCTION

The program of the Southern Utilization Research and Development Division is an organized effort through science and technology to increase present uses and to discover and develop varied new uses for Southern farm crops. Our farmers need new markets and strengthened demand for their production. At the same time, the Nation needs the new and better products that science can create from agricultural materials. To this end the Division conducts research on cotton, cottonseed, peanuts, tung fruit, citrus and subtropical fruits, peaches, rice, sugarcane, pine gum, replacement crops, sweetpotatoes, cucumbers and other vegetables.

The Division's program includes basic and applied research in the physical and biological sciences and engineering. Basic research plays a key role in uncovering new information that may be later exploited in applied research and development. When appropriate, engineers carry out pilot-plant studies of promising laboratory developments to provide engineering and cost data essential to industrial application feasibility determinations. The Southern Division has a total staff of about 554, and the in-house scientific effort in its research program amounts to approximately 281 professional man-years. The Division consists of two Pioneering Research Laboratories (Seed Protein and Plant Fibers), eight commodity-oriented Laboratories (Cotton Finishes, Cotton Chemical Reactions, Cotton Mechanical, Cotton Physical Properties, Oilseed Crops, Food Crops, Fruit and Vegetable Products, and Naval Stores), and one Laboratory (Engineering and Development) for engineering research and development. Headquarters of the Southern Division are located at the Southern Regional Research Laboratory, New Orleans, Louisiana. The Division also has personnel and laboratory facilities at Winter Haven and Olustee, Florida; Weslaco, Texas; Raleigh, North Carolina; Houma, Louisiana; and Natick, Massachusetts.

Division scientists consult with specialists from other organizations during both the planning and the execution of the research, and cooperate actively with industry to facilitate commercialization and utilization of new findings. Much of the cooperation is informal, but some work is conducted under conditions described in written cooperative agreements and memorandums of understanding. Currently 69 such agreements are in effect.

The farm products with which the Southern Division deals not only provide food, clothing and industrial raw materials, but also contribute to the Nation's general prosperity and well-being. Cotton, the Nation's number one cash crop, has an annual farm value of about $2.5 billion. The retail value of cotton products is about $18 billion. Cottonseed, a byproduct of cotton, has a farm value of around $300 million. The retail value of its products is about $1.8 billion. Citrus grown in the U. S. has a farm value

of about $500 million; vegetables over $1 billion; peanuts over $200 million; tung about $6 million; and gum naval stores about $30 million. The retail value of refined sugar produced from sugarcane grown in the United States and Puerto Rico is about $600 million. Industries processing these agricultural crops play a vital role in the Nation's economy; agribusiness today is about 30% of the Nation's total economy.

There is an urgent need for utilization research to help maintain the traditional food, feed and industrial outlets for agricultural products and to create new and larger markets for them. The opportunities are great; and, judging by past experience, the ability of utilization research to benefit the economy is tremendous. A recent example is the research development of stretch cotton products, whose commercialization has advanced rapidly to open up profitable new markets for cotton. These stretch products may represent the second major breakthrough in cotton processing (to give distinctly new products) within the last decade. The first was wash-wear cottons. Following are a few illustrative examples of significant developments based on the research of scientists at the Southern Division.

Commercialization of Stretch Cottons Advancing Rapidly. Consumer demand for stretch and bulked textiles in both wearing apparel and industrial applications is increasing rapidly. Research conducted by Southern Division scientists on imparting stretch and bulkiness to cotton products using various chemical and mechanical processes has contributed significantly toward opening up new markets for cotton in this area. The present annual minimum value of this research development is estimated at $2 million. At least nine finishers in the United States are in commercial production of cotton stretch fabrics by one of the processes (slack mercerization), and several other companies are preparing samples on a pilot-plant scale. Most of the production is going into apparel, but upholstery, slip cover material and coated fabrics are also being produced. Interest is also being shown in slack mercerized yarn for stretch knit goods. Two manufacturers of men's hose are producing experimental all-cotton stretch hose by slack mercerizing a very loosely knit hose. A number of companies are also investigating the possibility of producing cotton stretch products by several other approaches studied by Division researchers. Reliable estimates place the potential for stretch cottons at a figure comparable to that of wash-wear fabrics. Greater elasticity would make cotton a strong competitor in markets that by 1975 are expected to consume more than two and one-quarter million equivalent bales of all textile fibers in end-uses where this property is considered an important consumer quality.

Radically New Method of Carding Gains Wide Acceptance. The new SRRL Granular Card -- the first major change in the cotton carding machine since its invention 200 years ago -- is now widely used commercially. About 1000 Granular Cards are presently installed in the textile industry and others

are on order. Twenty-three manufacturers of textile equipment have been licensed by the Department to produce this device since its release to industry in 1959. The Granular Card reduces card waste by more than one-half and neps by about 10 percent. Flat strips are completely eliminated. With the device card processing costs are substantially reduced, operator health is benefited by the elimination of the major source of dust and fly in the card room, and yarn production is increased 1 to 3%. The new method of carding is especially advantageous for the production of coarse and medium number cotton yarns. The Granular Card promises a potential saving to the textile industry of about $2.2 million for each million bales of cotton processed through Granular Cards, assuming a sales price of twenty cents per pound for flat strips. This reduction in manufacturing costs adds to cotton's attractiveness for use in textiles.

Improved Cotton Batting Now Undergoing Commercial Evaluation. Excellent chemically-treated cotton batts having improved dimensional stability, coherence and resilience have been developed and produced experimentally on a pilot-plant scale by Division scientists in cooperative research with the National Cotton Batting Institute, Textile Waste Association, National Cottonseed Products Association, and the Foundation for Cotton Research and Education (affiliated with the National Cotton Council of America). Molded cotton batting products have also been made which appear promising for applications in head liners, bucket seats and crash pads in the automobile industry, and as padding in contoured furniture. These developments should make it possible for cotton batting to better meet the serious competition from polyurethane foams and foam rubber in padding applications and in mattresses. Two commercial plants have installed pilot lines for the production of the new cotton batting, and eight additional companies are contemplating the installation of pilot lines. Tests on auto seat cushions at two major automobile manufacturers have shown that the cotton batting at present stage of development substantially meets their requirements for cushioning materials. Sample mattresses made with the batting are currently undergoing evaluation.

New Fat Tempering Process Developed. A rapid tempering process for confectionery fats based on domestic products has been developed by scientists of the Southern Division and the National Confectioners Association in cooperative research. The process is based on the discovery that modification of fat crystals can be effected by mechanical working. The desired highest-melting crystal form of the fat, which is the form confectioners strive to achieve in conventional tempering processes, is produced rapidly by the new process and the tempering time of chocolate and similar fats can be reduced from several hours to a few minutes. Through use of this discovery, economies and improvements in the manufacture of confectionery fats and chocolate-type confections should be able to be accomplished. It should be possible to employ simplified and less expensive processing equipment; less material would be in process at one time; and better control of the tempering and coating processes should be achievable. The rapid tempering process has

received considerable industrial interest. One company is presently employing the process, and another commercial firm is evaluating it on a pilot-plant scale.

AREA NO. 1 - COTTON - BASIC AND EXPLORATORY INVESTIGATIONS

Problem. Cotton, the nation's most important fiber, is facing severe and increasing competition from synthetic fibers. Cotton is America's largest source of cash farm income and still accounts for almost two-thirds of the total U. S. mill consumption of all major fibers. However, its proportionate share of the market has been slowly decreasing as has the per capita consumption. The rapid growth of the synthetics at the expense of the natural fibers has been a phenomenon of the century. Expansion of market outlets for the chemical fibers has been based on vigorous research and development programs. The engineering and development programs of the chemical fiber industry are designed to capitalize on the special properties of each individual fiber as related to the functional use qualities desired in particular products; basically they involve the substitution of the newer fibers for cotton in cotton's traditional end use markets. Expanded research to increase the utilization potential of cotton offers the most realistic opportunity for improving cotton's competitive strength as a textile fiber and for increasing cotton consumption. Basic and exploratory investigations, studies on interrelations among fiber, yarn, and fabric properties, new and improved textile machinery, improvement of wash-wear properties and improved cotton properties and products are basic to holding existing markets or expanding the use of cotton in new applications.

Fundamental information is badly needed in applied research to help cotton gain new and maintain old markets. Fundamental knowledge of the cotton fiber as to its structure, properties, and the mechanisms involved in chemical and physical behavior serves as a basis and a guide in the design and improvement of processing machinery, mechanical and chemical processes, and in the development of new and improved cotton yarns, fabrics, finishes, and treatments. Many chemical and physical treatments, as well as textile organizations and machine designs, offer a basis for the improvement of cotton quality or lowering of processing costs. Exploratory chemical and physical research is needed to determine the true potential of such approaches prior to undertaking extensive developmental research or the construction of prototype machinery. Specific areas in which basic information is needed include the chemical properties and structure of native and modified cottons; the chemical modification of cotton cellulose; chemical reactions induced in cotton cellulose by high energy radiation; application of tagged elements to studies of native and modified cotton cellulose; reaction mechanisms, rates, and catalysis of cotton cellulose reactions; new concepts and methods for evaluating the physical properties of native and modified cottons; relationships of the structural arrangements within cotton fibers to the physical properties of native and modified cottons; mechanisms of physical damage to cotton due to mechanical, chemical, or biological actions; fine structural changes occurring during chemical and physical modification of cotton cellulose; and correlations of the fine structure of cotton fibers with their gross behavior in textile structures.

USDA PROGRAM

The Department has a continuing long-term program involving organic chemists, analytical chemists, physical chemists, physicists, microscopists, chemical engineers, statisticians, mathematicians, cotton technologists and textile technologists engaged in basic and exploratory studies to develop fundamental information needed in applied research to help cotton gain new and maintain old markets.

Basic research on the structure of cotton fiber and its relation to the behavior in mechanical and chemical treatments, essential to an understanding of the performance of fibers during processing and in textile products, is carried out at the Southern Regional Research Laboratory, New Orleans, Louisiana. Included is the research of the Plant Fibers Pioneering Research Laboratory to obtain basic information on the supermolecular structure of plant textile fibers; and to relate information of polymer and fiber structure to the mechanical and textile behavior of fibers. Additional basic research on chemical and physical properties and structure of cotton is being carried out under contract at Stanford Research Institute, South Pasadena, California, on determination of the structural components of the cotton fiber that contribute most to tensile strength and how they can be utilized to increase tensile and recovery properties, and at Texas Agricultural Experiment Station, College Station, Texas, on the effect of variation in structure on cotton fiber properties caused by environmental and genetic factors. Exploratory chemical and physical research is also conducted at New Orleans, Louisiana, as a basis for the improvement of mechanical and chemical processing, and in the development of new and improved yarns, fabrics, finishes, and treatments. Additional exploratory chemical and physical investigations are being carried out under contract, at the Massachusetts Institute of Technology, Cambridge, Massachusetts, on the mechanics of nep formation in cotton during processing, and at General Aniline and Film Corporation, New York, N. Y., on the reaction of acetylene and related compounds with cotton cellulose.

Other research on chemical and physical properties and structure of cotton is in progress under grants of P.L. 480 funds to the following foreign institutions: Ministry of Commerce and Industry, Jerusalem, Israel, for fundamental investigation of crimp in cotton fibers and its relationship to other fiber properties (project duration - 5 yrs.); National Institute of Applied Chemical Research, Paris, France, for a fundamental study of the relation of crystallinity to accessibility in cottons (project duration - 5 yrs.); Swedish Institute for Textile Research, Goteburg, Sweden, for an investigation of setting reactions in cotton fabrics (project duration - 5 yrs.); Central Laboratory, T.N.O., Delft, Holland, for a fundamental study of the response of cotton fiber structural elements to stress (project duration - 3 yrs.); Ahmedabad Textile Industry's Research Association, Navrangpura, Ahmedabad, India, for a study of the relation between fine structure and mechanical properties of cotton fibers by swelling and stretching treatments (project duration - 5 yrs.); The Cotton Silk &

Man-Made Fibres Research Association, Shirley Institute, Didsbury, Manchester, England, for a fundamental study of the microbiological breakdown of cotton fiber (project duration - 5 yrs.); University of Bombay, Bombay, India, for an investigation of the photochemical degradation of cotton (project duration - 5 yrs.); and for an investigation of new solvents for molecular weight determination of cellulose (project duration - 3 yrs.); and Technological Laboratory, Indian Central Cotton Committee, Bombay, India, for investigation of the microbial decomposition of cellulose with special reference to the effect of Indian bacterial organisms on cotton and cotton fabrics (project duration - 4 yrs.). Exploratory chemical and physical investigations are in progress under grants of P. L. 480 funds to the following foreign institutions: The Cotton Silk & Man-Made Fibres Research Association, Shirley Institute, Didsbury, Manchester, England, for a fundamental study of the pyrolysis of cotton cellulose (project duration - 5 yrs.); and Birkbeck College of University of London, London, England, for a fundamental investigation of preparation and properties of certain phosphonitrilic derivatives for use in treating cotton (project duration - 4 yrs.); and for a fundamental study of the preparation and properties of phosphazene and phosphoryl chloride derivatives having potential for reaction with cotton cellulose (project duration - 4 yrs.).

The Federal in-house scientific effort devoted to research in this area totals 51.6 professional man-years. Of this number 27.0 is devoted to chemical and physical properties and structure and 24.6 to exploratory chemical and physical investigations. The contract research involves an additional 6.4 man-years, 2.8 being on chemical and physical properties and structure, and 3.6 on exploratory chemical and physical investigations. P. L. 480 research involves 12 grants, of which 9 are on chemical and physical properties and structure and 3 on exploratory chemical and physical investigations.

The following lines of work were terminated during the year: (1) A study of the etherification of cellulose using radioactively labeled etherifying reagents, and (2) A microscopical study of degradation of cotton cellulose structure by various agents (under chemical and physical properties and structure); and (3) Crosslinking of cotton cellulose with difunctional etherifying agents using alkaline catalysts, (4) Systematic exploratory investigation of chemical pretreatments as a means of producing resilient cotton fabrics having improved abrasion and tear resistance, (5) Exploratory study of means of producing thermoplastic cottons, and (6) Fundamental study of the effect on cotton of oxidizing agents (P. L. 480 project), (under exploratory chemical and physical investigations).

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical and Physical Properties and Structure

1. Application of Radioactively Labeled Reagents to Studies of Native and Modified Cottons. The basic studies of the etherification of cellulose using radioactively labeled reagents have been terminated. A method was developed for methylating cotton cellulose, which has the same chemical and radiochemical analysis, for use in studying reactions of methylated cotton cellulose. Indications are that it would be feasible to employ this type technique in connection with other chemical modification processes for cotton, including reactions involving di-functional etherifying agents. The technique may make possible, for the first time, definitive investigations of the kinetics and reaction mechanisms of chemically modified cotton celluloses of potential importance to cotton utilization. The stability and susceptibility of modified celluloses to deterioration and degradation by acids, alkalis, selected solvents, oxidizing agents, heat, both photochemical and high energy radiation in the presence or absence of reducing and oxidizing reagents, and micro-organisms can be investigated. (S2 1-151).

2. Studies of New Concepts and Methods for Evaluating the Physical Properties of Native and Modified Cottons. Research was continued to evaluate the swelling behavior of cotton fibers in various environments by observation of the untwisting of single yarns. Preliminary evaluation of the swelling behavior of both mercerized and scoured yarns in thirty-three selected liquids by observation of the untwisting rate of the yarns indicated that classification of the liquids into groups of zero, low, intermediate, and high swelling characteristics is possible. All of the alcohols studied fell into the intermediate group, but members within other classes of compounds did not fall into a common swelling group. A close correlation was obtained between the yarn untwisting measurements and diameters of the fibers as measured by a microscopic and projection technique. When the study was extended to water dilutions of five alcohols (methanol, ethanol, propanol, t-butanol, isopropanol), bases (sodium and potassium hydroxides), an amine (ethanolamine), and an aldehyde (formaldehyde), both the mercerized and scoured yarns showed similar trends in swelling behavior in most cases. Of the groups of liquids tested, the alcohols proved to be the most similar within a given class. Should this untwisting method prove valid, it could provide a quick and useful technique for determining the ability of cotton to imbibe reagents used in the chemical finishing of cotton textiles. (S2 1-182).

3. Basic Studies of the Relationships of the Structural Arrangements Within Cotton Fibers to the Physical Properties of Native and Modified Cottons. Valuable information on the structure of the fiber at the microfibrillar level has been obtained by treatment of fragments of modified cottons with various reagents while on specimen grids of the electron microscope, followed by microscopic examination. Results on cyanoethylated cotton crosslinked with DMEU confirm the general conclusion, previously

reached with acetylated cottons, that crosslinking makes the fiber more susceptible to fracture. Removal of cyanoethyl groups from the cellulose and hydrolysis of the DMEU allows cellulose to return to a normal structure at the microfibrillar level. As might be anticipated, removal of the cyanoethyl group from cyanoethylated cotton with 25% NaOH resulted in a different type of breakdown than that observed when acetyl groups were removed from acetylated cotton by acid hydrolysis; hydrocellulose particles were not obtained when the cyanoethyl groups were removed, but swelling and dissolution were pronounced. Fragmentation patterns have also been obtained on a series of benzylated cottons. Studies of cotton fibers in which styrene has been polymerized by gamma radiation point to the possibility of graft polymerization within the layers of the fiber wall. The application of dyes containing heavy metals, for example sulfone type dyes with chromium, appears promising as a means of investigating accessible sites within cotton and modified cottons and will be investigated further. (S2 1-174).

The effect of gross and fine structure on the physical behavior of cotton fibers is being studied in recently initiated research. A complete understanding of the relation of the unique physical properties of cotton to the structural features is a vital step in selecting fibers for end uses, constructing and treating fabrics for best use, and in breeding. It has been found that examination of uncollapsed fibers from undried immature bolls with a polarizing microscope gives enhanced observation of the spiral structure of cotton. The reversing helical structure is clearly evident, and upon further swelling of the undried fibers in zinc chloride solution it is apparent that the outer layers responsible for balloon formation usually spiral in the same direction as the gross thickening. Plans have been made to grow cotton fibers under controlled conditions by contract in cooperation with Crops Research Division. Other cottons of unusual structural features will be sought from various sources for the research. (S2 1-208).

Fundamental studies of the role played by the structural elements of the cotton fiber in response to stress are being conducted in P. L. 480 research at the Central Laboratory, T.N.O. Through the use of modern microtechniques for manipulating and observing single fibers, a better understanding is being obtained of the internal movements that occur within the cotton fiber while it is being subjected to stretching. Progress is being made in obtaining basic knowledge of cotton fibers that eventually will be directed toward efforts to improve cotton fiber properties through cotton breeding programs and improvements in cotton processing. (UR-E19-(20)-4).

In P. L. 480 research at the Swedish Institute for Textile Research, reactions which will cause setting in cotton fabrics and garments are being investigated. Treatment of cotton fabrics with solutions of certain inexpensive alkalis or inorganic salts has been shown to cause the relaxation of internal stresses in the fabrics. This treatment, which is generally known as "setting," decreases surface mussiness of the fabric, and in combination with standard resin treatments, results in improved wash-wear properties. Progress is being made toward providing the basis

for reducing the amount of resin required to provide acceptable wash-wear qualities in cotton textiles. (UR-E26-(20)-2).

4. Elucidation of Mechanisms of Physical Damage to Cotton Due to Mechanical, Chemical, Physical or Biological Actions. Fundamental studies of the microbiological breakdown of cotton are being conducted at the Shirley Institute under a P. L. 480 research grant now nearing its expiration date. Cotton fabrics in many applications are exposed to bacteriological and fungicidal attack. Studies of the enzyme systems involved have shown that these systems consist of at least two types. One of these, less of which is present in isolated enzyme preparations, appears to penetrate the cellulose more readily, thus accounting for the significantly greater degradation of cotton known to be caused by living organisms than by isolated enzyme alone. Knowledge of the mechanism by which microbiological breakdown occurs will assist in the design of more efficient rot-proofing and mildew-proofing treatments for cotton products. (UR-E29-(20)-6).

An investigation of the photochemical breakdown of cotton under different conditions of exposure to radiation is being carried out in P. L. 480 research at the University of Bombay. It is well known that cotton fabrics are weakened by prolonged exposure to sunlight or to strong illumination. Progress is being made toward determining the mechanisms by which photosensitization and photolytic degradation of cotton and selected modified cottons take place. Basic knowledge of the reaction mechanism and kinetics is expected to be useful in devising practical means to prevent the deterioration of exposed cotton fabrics by means of chemical inhibitors or screening agents that prevent or interfere with the sequence of reactions involved. (UR-A7-(20)-4).

5. Investigation of the Structural Changes Occurring During Chemical and Physical Modification of Cotton Cellulose. Work on fine structure of plant fibers has been pursued along a number of lines by the Plant Fibers Pioneering Research Laboratory. The objective of this Laboratory is to obtain basic information on the supermolecular structure of plant textile fibers; and to relate information of polymer and fiber structure to the mechanical and textile behavior of fibers. Several lines of investigation were concluded during the year and the results are being prepared for publication.

A new line of work, initiated during the year, seeks to examine by means of X-ray line broadening the crystallite size and size distribution in plant fibers, a subject considered to be of no little significance. Since the prior work in this area has been negligible, the first approach has necessarily been directed along the lines of methodology. The instrumental contribution to line broadening has been found to be minor. Model substances examined thus far have consisted of ultrafine quartz powder, reduced by vibration ball milling, and amorphous sugars such as glucose and cellobiose, prepared by the process of lyophilization.

The work on thermal properties and behavior of highly acetylated cotton fiber cellulose has been completed. In the concluding studies it was shown that heat crystallization of the acetylated yarn, which occurs at 175-200°C., leads to losses of strength of roughly 20%, as compared with the uncrystallized yarns though the products were still equal in strength to the unacetylated controls. Crystallization increased the extensibility of the yarn somewhat, especially at the higher degrees of substitution. Heat crystallization greatly improves the elastic and work recovery behavior of the acetylated yarn between 100-200°C. as compared to uncrystallized controls. Whether the excellent properties of highly acetylated cotton will make its preparation commercially advantageous remains for economic considerations to determine.

Studies of the thermoplastic behavior of highly acetylated yarns, which have been concluded, showed the strong effect on heat-setting capacity of the degree of substitution, temperature and steam plasticization.

Progress in the research on modification of cellulose structure by chemical substitution has been retarded because of the need for designing and constructing a suitable reaction apparatus. Such equipment was eventually evolved through the development of a rather generally applicable assembly in which all parts exposed to the reactant, including the circulating pump, are either coated with Teflon or are constructed of Teflon, nylon or glass. Techniques have been improved, and, particularly, a means has been provided for applying controllable tension on the yarn during reaction. Preliminary results show a favorable degree of uniformity of the reacted yarn. A second method, less fully explored, consists in reacting short lengths of knit tubing which can be unravelled after reaction.

Study of the changes in fine structure of cotton fiber with progressing fiber development, which has been dormant for most of the year, has been resumed on freeze-preserved samples. Preliminary accessibility results appear to be comparable to those obtained earlier on alcohol-preserved samples.

Investigations of amine coordination compounds of cellulose have been brought to a close. Studies of cotton and rayon celluloses, heated in the presence of ethylenimine and acetic acid, indicated that aminoethyl groups are attached to the cellulose in the amorphous regions, and the crystal lattice of the celluloses remains intact. Metal ions such as copper are readily complexed by the treated material.

Attempts to prepare cellulose I in single crystal form in the laboratory have been delayed because of difficulty in preparing suitable cellulose triacetate single crystals necessary for saponification to the desired polymorphic form.

An improved infrared technique for determining crystallinity of cellulose--applicable to celluloses I and II, or their mixtures--has been developed. This procedure should furnish a better and more rapid means of assessing

the degree of crystallinity of cellulosic materials exposed to various treatments, such as mercerization, heat, radiation and the like.

Other in-house research (not in PF Pioneering Research Laboratory) involves investigation of improved infrared spectral techniques for the study of modified cottons to evaluate molecular structural changes produced by chemical and physical treatments. The infrared spectral analysis of chemically modified cottons, particularly those reacted with crosslinking agents, will permit identification of cotton cellulose derivatives which have been difficult to characterize in detail by chemical methods. In recent work, the use of spectra of the acid hydrolyzates of chemically modified cottons has been investigated and appears to be a valuable adjunct to the conventional and differential spectral methods previously developed. A scheme of analysis employing a combination of conventional, differential, and acid hydrolyzate spectra has been successfully applied to a variety of resin-treated cotton products. The hydrolyzate spectra show a certain degree of variability, but in general are characteristic of the derivative examined. This approach offers a potentially powerful means for studying the type and extent of reaction in chemical modifications which proceed to relatively low degrees of substitution. The research will be continued with emphasis on the use of the differential spectral technique to reveal new infrared absorption bands in cotton derivatives and hydrolyzates. (S2 1-165, S2 1-220).

Research has been initiated on the separation and identification of the cleavage products of partially etherified cottons, including crosslinked cottons, as a possible basis for elucidating the chemical structure of the modified celluloses, including the position of the substituents or crosslinks on the glucose unit and in the fiber structure. Hydrolysis of aminoethylated cotton followed by isolation of a nitrogen-rich fraction has been achieved. The fraction contains several materials which have been tentatively identified as aminoethyl-sugars. Another approach for cleaving chemically modified cottons--oxidation of the cellulose to the dialdehyde form, reduction to the dialcohol form, and subsequent hydrolysis --has been explored in limited experiments on cotton cellulose with encouraging results. Both hydrolysis and the latter approach will be applied to selected etherified cottons. The information developed should prove helpful in guiding research on the development of new cotton textiles having properties that will enhance their commercial value. (S2 1-214).

In P. L. 480 research at the National Institute of Applied Chemical Research, a basic study of the fine structure of the cotton fiber is being conducted in an effort to relate the fine structure to other fiber properties that are important in the processing and use of cotton. Improved methods, both physical and chemical, have been devised for measuring differences in the fine structure of cottons. These have been applied to a typical U. S. cotton of Deltapine variety, the fiber properties of which have been extensively studied in several laboratories, and are thoroughly known. These studies are now being extended to a series of raw, purified

and chemically crosslinked cotton yarns, all spun from the same Deltapine cotton. Three commonly used crosslinking treatments, all easily measured by analytical procedures, are included in this phase of the investigation. The information obtained eventually will be translated into the development of improved cotton products. (UR-E9-(20)-61).

6. Relationship of Cotton Fiber Gross Structure to Behavior of the Fibers in Textile Structures. A fundamental investigation of the interfiber frictional force and associated fiber properties has been initiated to improve the processing of cotton products. The research is based largely on leads developed in earlier work on effect of fiber drag on the draftability of cottons.

Samples of cotton differing extremely in fiber properties and those exhibiting unusual "spinning" properties are being accumulated as a step in measuring their frictional and fiber drag properties. Methods for measuring fiber friction and torsional rigidity are being investigated. In a large-scale ends-down experiment on a medium staple cotton, it has been found that the greater the amount of fiber hooks leading into a process, the greater the adverse effect on sliver and roving uniformity, yarn strength and end breakage in spinning. Yarn strength can be moderately improved and end breakage can be substantially improved by selecting the drafting procedure which best reduces the incidence of fiber hooks. By using the optimum procedure spinning speeds can be increased 33 percent over that using the conventional procedure while maintaining an equivalent ends-down rate. The effect of fiber hooks in short and long staple cottons will be investigated. (S2 1-201).

A fundamental investigation of fiber crimp, a property possibly responsible for differences in mechanical processing behavior of cotton fibers, is in progress under P. L. 480 research at the Ministry of Commerce and Industry of the State of Israel. An optical projection system was developed to measure crimp in two perpendicular planes. The apparatus has now been improved through the development of a special curve tracer in conjunction with an electronic computer that permits the continuous and dynamic measurement of the crimp diameter, which is considered to be the main crimp parameter of cotton fibers. Crimp diameter decreased with tension for Deltapine 15 fibers. When energy required to uncrimp fibers was measured, that for Deltapine fibers remained essentially constant after the first stretching cycle. Energy for Acala 1517 fibers decreased with the first through fourth stretching cycles and increased with period of relaxation, a distinct difference in behavior of these two cottons. These improved techniques for evaluating fiber crimp are being applied to a number of cottons of differing physical characteristics. (UR-A10-(20)-5).

B. Exploratory Chemical and Physical Investigations

1. Exploratory Chemical Modification of Cotton Cellulose. A new method for the chemical modification of cotton cellulose may result from the

discovery that acetylenic compounds such as propiolic acid and acetylene dicarboxylic acid react under strongly alkaline conditions to crosslink cotton cellulose and introduce carboxyl groups at the same time. In other research, bis(chloromethyl)sulfone has been successfully reacted with cotton in anhydrous media by the use of sodium cellulosate. This reaction could not be achieved when alkali cellulose was employed. It should be possible to prepare other new derivatives of cellulose using the sodium cellulosate approach. (S2 1-161).

Several approaches have been followed in research to chemically attach reactive compounds to cotton cellulose by means of polyfunctional reagents. A variety of hydroxy, phosphorus and sulfur compounds, and certain dyes, have been attached to cotton with glyoxal using magnesium chloride as catalyst. Treated fabrics showed improvement in dry and wet crease recovery, permanent starching, and permanent dyeing. Glyoxal is similar to formaldehyde as a bonding agent, but has the disadvantage of causing discoloration.

Exploratory experiments have also indicated that treatment of cotton cellulose with selected sodium salts of organic and inorganic acids and formaldehyde may offer a method for imparting increased wet and dry crease recovery, accompanied by breaking and tear strength values that are higher than those obtained with formaldehyde alone.

Further progress in eliminating the need for afterbleaching of sulfone-treated fabrics has been made. The yellowing that occurs during the commercial treatment of cotton with bis(hydroxyethyl)sulfone has been found to be due to degradation products of cellulose formed by heat and the alkaline catalysts, as well as to yellowing of polyethylene softeners by oxidation. This important finding has been shown to be applicable to yellowing in other base-catalyzed heat-curing processes, such as aminization and chemical treatment of cotton with APO. The yellowing has been eliminated for sulfone treatments at curing temperatures up to 180° C. by use of combinations of borax and sodium borohydride, obviating the need for an afterbleach. As little as 0.01% of sodium borohydride, in combination with borax and a fluorescent brightener, prevented the yellowing normally produced by cotton and also by a polyethylene softener, during high-temperature (180° C.) curing with bis(hydroxyethyl)sulfone. The low level of sodium borohydride required would make its use less expensive than the afterbleaching step currently being practiced.

In another phase of work, which is presently being emphasized, a number of vinyl polymers were crosslinked on cotton when cured with benzoyl peroxide to give unusual coatings which leave the fabric pliable and permeable to air, even at weight gains of 30%. Considerable water repellency and abrasion resistance were imparted, and the coatings were not extractable by organic solvents. Free-radical crosslinking of polymers on cotton appears to be a new field of cotton finishing, and offers great promise of giving cotton products with increased resistance to light, microorganisms and weathering, as well as improved water repellency. (S2 1-186).

Investigations of pretreatments to improve the physical properties of chemically modified cotton textiles have continued. A combination of pretreatments (consisting of treatment of slack yarn with 70 percent nitric acid, restretching, and mercerization at constant length) gave, possibly for the first time, a cotton yarn showing no loss in tensile strength when crosslinked with DMEU. A cotton fabric having considerable luster, high dry wrinkle resistance (279° W+F), fair stretch (21% elongation at break) and very high tearing strength was obtained by mercerizing kierboiled yarn under tension at 3% stretch, weaving the pretreated yarn into 90 x 42 broadcloth, and crosslinking the woven fabric with DMEU. Preliminary findings also make it appear that fabric woven of grey yarn pretreated by mercerizing under moderate tension and restretching, with or without scouring in fabric form followed by crosslinking with DMEU, shows little change in tearing strength during the crosslinking step itself. The breaking strength decreases, however. Exploratory work will continue under a new project on the crosslinking of various physically modified crystalline forms of cotton as a means of producing resilient cotton textiles having improved appearance and durability to wearing. (S2 1-167).

Improved methods of etherification of cotton cellulose with benzyl chloride have been developed. Optimum time allowed for premercerization was established to be 30 minutes; use of potassium hydroxide in place of sodium hydroxide increased the benzylation rate 50%; and alkali metal iodides, such as those of sodium and potassium, produced a twofold-to-threefold acceleration of the reaction rate. The catalytic effect of iodide ion is attributed to its reaction with benzyl chloride to yield benzyl iodide and chloride ion. The benzyl iodide is believed to be much more reactive towards cotton than is benzyl chloride. The rate of benzylation, employing benzyl chloride and alkali concentrations of maximum swelling, was found to be 3-4 times as great using rubidium or cesium hydroxide as when using lithium hydroxide. The extent of formation of cellulosate ions, rather than the degree of swelling, evidently controls the benzylation rate of cotton. In studies of the reaction of 1,4-bis(chloromethyl) benzene with cotton yarn, the p-xylylene group (which has nearly the same molecular size, shape and weight as the benzyl group) failed to impart thermoplasticity in contrast to the latter group, apparently because it was attached as a crosslink. Leads developed in this exploratory study of means of producing thermoplastic cottons will be applied in new research on improved methods of etherifying cotton cellulose. (S2 1-172).

The exploration of cellulosic crosslinks capable of being broken and reformed at will has continued. Reaction of acethydrazide disulfide with dialdehyde cotton fabric increased both wet and dry wrinkle recovery. However, reduction to break disulfide crosslinks, followed by oxidation in the swollen state to reform them, did not successfully restore dry wrinkle recovery. Evidence was obtained indicating that the disulfide was predominantly attached monofunctionally rather than in a crosslink. In other experiments, cotton was crosslinked with the iodine oxidation product of 3-chloro-2-hydroxypropyl mercaptan, presumably the disulfide. These

crosslinks could be broken by reduction and then easily reformed by mild oxidation, even air-oxidation. It has also been shown possible to use the azo linkage as an easily broken and reformed portion of cellulose crosslinks. Cotton crosslinks obtained with an azo-group containing reactive dye (Remazol Black B) can be broken by reduction. Diazotization of remaining amino groups, followed by coupling with aminonaphthols, restores the crosslinks. Preparation of a difunctional azo-containing compound by reaction of acrylyl chloride and p-phenylenediaminesulfonic acid, followed by oxidation, is under investigation. Compounds of this type will be synthesized for use in introducing crosslinks containing azo groups into cotton. (S2 1-168).

The overall objective of completed fundamental investigations of the reaction of cotton with epoxy compounds was achieved in that cotton was reacted with mono- and polyepoxides in the presence of free radicals and other disproportionation products as catalysts, cellulosic reactions were initiated photochemically, and physico-chemical techniques were used to elucidate cellulose-epoxide reaction mechanisms. Fundamental information was developed on the application of d,l-butadiene diepoxide, its dihalohydrin precursors, and epichlorohydrin to cotton in the presence of various concentrations of base and in the presence of basic solutions concentrated with respect to various salts. Cottons having dry as well as wet crease resistance were obtained by pretreating with sodium hydroxide solutions (2 to 23% concentrations) and reacting with the d,l-butadiene diepoxide. The rates of acid and base catalyzed hydrolyses of various diepoxides have been determined from 25° to 90° C. as a basis for improving the efficiency of cellulose-epoxide reactions. The fabric properties imparted to cotton by diepoxides have been shown to be durable to acid and alkaline washes, and are not damaged by chlorine-scorch tests. (S2 1-169).

Contract research has been initiated at General Aniline and Film Corporation to conduct exploratory investigations of the reaction of acetylene and related compounds with cotton cellulose. The objective is to chemically modify cotton cellulose and selected modified cottons using these agents, and determine the chemical and physical properties of the products to ascertain their potential for various end-uses. (S2 1-199(C)).

New research is in progress to gain fundamental knowledge of the influence of selected lead compounds on cotton, and to impart special properties to cotton through application of lead compounds. Preliminary work indicates that the precipitation of insoluble lead compounds by the two bath method is apparently a simple but effective way for applying lead to cotton. Numerous lead derivatives have been screened in this manner but the treated cotton samples have not been completely evaluated as yet. The chloride, hydroxide, and esters of triphenyl lead show promise of being modified to become cellulose reactive and thereby impart special properties to cotton. Both of these research approaches are being continued. (S2 1-202).

The preparation and properties of compounds of unusual chemical structure

composed of phosphorus and nitrogen are being investigated at Birkbeck College, University of London, under a P. L. 480 grant now nearing its expiration date. Basic and fundamental information to put the chemistry of these inorganic compounds on a sound systematic basis, similar to that for carbon chemistry, has been sought. New methods of synthesis have been devised, and improved methods of separation and purification have been worked out. Employing these, entirely new compounds have been discovered and previously known derivatives improved through purification, resulting in compounds of altered properties and greater thermal stability. Some of the compounds have potential as reactive finishes to confer properties such as wash-wear and flame resistance to cotton fabrics. (UR-E29-(20)-35).

2. Chemical Reactions Induced in Cotton Cellulose and Chemically Modified Cotton by High Energy Radiation. Thermoplastic graft polymers of cotton have been made by use of simultaneous irradiation of vinyl monomers (acrylonitrile or styrene) and cotton cellulose or cyanoethylated cotton with high energy gamma radiation to initiate the graft polymerization reaction. In studies of the physical properties of these graft polymers it was found that the summations of the relative energies expended over a temperature range from 21° to 200° C. to elongate various fibrous polymers to 1.5% were as follows: cyanoethylated cellulose (D.S. 0.7) >polyacrylonitrile-cellulose>cellulose>polystyrene-cellulose>polyacrylonitrile-cyanoethylated cellulose. Exploratory investigations to determine the molecular weights of the graft polymers of polyacrylonitrile-cotton cellulose, polystyrene-cotton cellulose, and polyacrylonitrile-cyanoethylated cotton cellulose and to relate these molecular weights to the properties of the fibrous products have been initiated. Several fabrics, chemically modified by various processes developed at the Southern Division, were irradiated, and selected properties of these products were determined. No improvements in properties were noted for those studied thus far. (S2 1-176).

New research was initiated on investigation of the energy yields of the gamma radiation-induced reactions of cotton. The reactions of the long-lived free radicals produced in cotton by gamma radiation were investigated by studies on the initiation of graft-polymerization of acrylonitrile, methyl methacrylate and vinyl acetate onto cotton fibers, yarns and fabrics after removal of the cotton from the radiation source. The results obtained could be explained in terms (1) of the production of long-lived free radicals in the accessible regions of the cellulose (in an inert atmosphere) and the inaccessible regions of the cellulose structure (irrespective of atmosphere) and (2) of the penetrating ability of the treating solution. Vinyl acetate reacted much slower than the other two monomers. The polyacrylonitrile-cotton yarns were thermoplastic, and polyacrylonitrile-cotton fabrics had increased flex and flat abrasion resistances as compared with untreated fabrics. Further evaluation of the mechanical properties of vinyl monomer grafted yarns and fabrics, prepared by the post-irradiation technique, will be carried out. Work on the grafting of ethylene to cotton by the post-irradiation technique will be initiated. (S2 1-195).

3. Basic Investigation of Reaction Mechanisms, Rates and Catalysis of Cotton Cellulose Reactions. In initial work on a fundamental study of mechanisms of cellulose etherifications, the rates of reaction between cotton and a selected N-methylol etherifying agent have been measured. Also, the infrared spectra and X-ray diffraction patterns of cottons etherified with an epoxide in the presence of various concentrations of aqueous sodium hydroxide (2-23%) are being interpreted in an effort to correlate changes in crease recovery properties with changes in fine structure. Suitable spectrophotometric or electrometric methods of detecting transition complexes between the catalyst and etherifying agents are being sought. (S2 1-196).

P. L. 480 research at the Shirley Institute on the pyrolysis of cotton cellulose is continuing to provide information needed for improvement of flame-resistant treatments for cotton. Reduced flammability of cotton textiles for apparel, draperies, awnings, etc., is highly desirable from the standpoint of safety. The nature of the reactions involved in the burning of cotton are being studied in three different experimental systems designed to give information on what reactions occur when cotton is heated under various conditions and at different rates, and the sequence of these reactions. It has been found that the first products of pyrolysis (oxygen-containing materials such as tars, carbon monoxide, etc.) require little outside oxygen for further combustion and that the degree of flame resistance of cotton fabrics is related to the char to tar ratio upon pyrolysis. This knowledge indicates that fabric finishes designed to reduce the flammability of cotton should be directed toward altering the sequence of these reactions to prevent the formation of combustible products. Certain phosphorus-containing permanent textile finishes have been shown to function in this manner. (UR-E29-(20)-9).

Research under a P. L. 480 grant, recently expired, has been completed at the Ministry of Commerce and Industry of the State of Israel on a fundamental study of the oxidation of cotton by various oxidizing agents to obtain data on the kinetics of the oxidation and the changes in physical and chemical properties which occur. Hypochlorite is widely used in the finishing of cotton textiles. Basic investigations of oxidation of cotton with hypochlorite, the common commercial and home bleaching agent, have explained the mechanism of the degradation which occurs. The results of this research indicate oxidation and resulting breakdown is random and involves both cleavage of the cellulose polymer chain followed by oxidative attack at these points. These degradative changes are a function of oxygen consumption rather than the pH of oxidation. The more intimate knowledge of the mode of action of commercial bleaching agents gained in this research should lead to improvements in their application, both in the laundering of clothing to provide longer service life, and in improved products in cotton textile finishing. (UR-A10-(20)-4).

4. Exploratory Physical Investigations on Cotton. Experimental findings thus far in the contract research at Massachusetts Institute of Technology

on mechanics of nep formation in cotton indicate that perhaps too much blame is being placed on textile processing machinery for the nep increases observed during mechanical processing. It appears that the harvesting and ginning processes are responsible for the relatively large neps present in the cotton received by the textile mill. Many of these neps break down into smaller ones as the cotton goes through various stages of textile mill processing, increasing the total number of neps in the processed cotton. A textile process simulating device has been redesigned to facilitate the taking of motion pictures of neps as they are formed. The new unit is now ready for testing. Continued photographic studies of the flow of air and fluids around models of card wires, both fillet and metallic, show areas of turbulence which are highly conducive to fiber tangling, hence nep formation. The drafting process and its effect on the removal of fiber hooks and on the further entanglement of fibers under the influence of the drafting forces are also under investigation. Theoretical considerations, using principles analogous to those employed in the study of configurational versatility of long polymer chains, link nep formation to such physical properties as Young's Modulus, Micronaire Reading, and Causticaire Maturity Index. (S2 1-173(C)).

Continued progress has been made in fundamental studies of the drying of chemically treated cotton fabrics as a basis for improving the efficiency of textile drying and curing operations. Drying rate experiments with resin-treated cotton poplin indicate that resin migration can be quantitatively detected by a technique involving measurement of the change in drying rate resulting from deposition of resin in the internal pore spaces of the fiber. This should prove helpful in development of one-step drying and curing techniques. Analysis of the data also demonstrated that other morphological characteristics of the fabrics can estimate closely the optimum pick-up to uniformly wet the interior without leaving localized excesses or deficiencies of treating material. Drying rate curves obtained for a series of partially acetylated cotton fabrics showed that drying rates for this type of chemically modified cotton correlated more closely with fabric thickness than any other variable studied. Additional theoretical aspects will comprise electronic computation of the drying rate data to ascertain the effect of capillary action in drying by comparing experimental curves with those electronically calculated for purely diffusional mechanisms. The next experimental work will relate principally to checking of previous results by duplication tests and extension of tests to include fabrics of different construction. (S2 1-188).

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AREA NO. 2 - COTTON - INTERRELATIONS AMONG FIBER, YARN, AND FABRIC PROPERTIES

Problem. The intense competition in today's textile markets is placing increasing demands upon cotton producers and processors for high quality products tailored to meet specific use requirements. Improvement in the quality of processed products and lower costs of mechanically processing cotton into yarns and fabrics are needed to satisfy consumer demands and maintain cotton markets. For example, information is needed to determine the effect of the important fiber properties and combination of fiber properties of cottons on yarn and fabric properties and processing performance to obtain the maximum utilization potential from cottons of different fiber properties and to provide guidance for cotton breeders in developing strains having more desirable fiber properties. Improved mechanical processing methods are needed to attain maximum yarn uniformity and the resultant improvements in the general quality level and processing efficiency of all types of cotton products. New and improved methods and instruments for measuring the physical and chemical properties of cotton are needed to guide processing research in developing new and improved products.

USDA PROGRAM

The Department has a continuing long-term program involving cotton technologists, textile technologists, textile engineers, physicists, statisticians, and mathematicians engaged in research to develop fundamental information and improved processing procedures in order to improve the quality and lower the cost of cotton products during the mechanical processing of cotton fibers into yarns and fabrics.

Research to determine the effect of fiber properties on processing efficiency and product quality is carried out at New Orleans, Louisiana. Additional research of this type is conducted under contract at Auburn Research Foundation, Inc., Auburn, Alabama, involving large-scale spinning evaluations of the effect of fiber properties and spinning variables on yarn properties and end breakage. Cooperation is maintained with cotton merchants and textile mills; the Crops Research Division, specially on the procurement of cotton of known history with special fiber properties; and the Market Quality Research Division, AMS, to insure coordination of effort in related research. Research on development of new and improved methods and instruments for measuring the physical and chemical properties of cotton, and evaluating the processing characteristics of cotton, is carried out at New Orleans, Louisiana.

Other research on effect of fiber properties on processing efficiency and product quality is in progress under grants of P. L. 480 funds to the following foreign institutions: The Cotton Silk & Man-Made Fibres Research Association, Shirley institute, Didsbury, Manchester, England, for a

fundamental investigation of the causes of warp breakage in the weaving of cotton yarns (project duration - 5 yrs.); and Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for an investigation of the relationship between the cohesion of cotton fibers and the properties of rovings and yarns (project duration - 4 yrs.), and for an investigation of the effect of drafting force on cotton yarn strength and uniformity (project duration - 5 yrs.). Research on development of new and improved methods and instruments for measuring physical properties of cotton is in progress under grants of P. L. 480 funds to the following institutions: Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for the development of methods and equipment for determining irregularity of transparency of card web and for counting neps (project duration - 4 yrs.); and German Research Institute for Textile Industry, Reutlingen-Stuttgart, West Germany, for the development of an apparatus for counting neps in cotton card web (project duration - 4 yrs.).

The Federal in-house scientific effort devoted to research in this area totals 15.2 professional man-years. Of this number 14.6 is devoted to investigations of effect of fiber properties on processing efficiency and product quality and 0.6 to development of new and improved methods and instruments for measuring the physical properties of cotton. The contract research involves an additional 0.9 man-year, all of the effort being on investigation of effect of fiber properties on processing efficiency and product quality. P. L. 480 research involves 5 grants, of which 3 are on effect of fiber properties on processing efficiency and product quality and 2 on development of new and improved methods and instruments for measuring physical properties of cotton.

The following line of work was terminated during the year: (1) Determination of the effect of the principal types of spotted cotton on product quality and processing efficiency to obtain optimum use of such cotton (under effect of fiber properties on processing efficiency and product quality).

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Effect of Fiber Properties on Processing Efficiency and Product Quality

1. Effect of Cotton Fiber Properties Such as Length, Strength, Fineness and Elongation on Fabric Properties and Processing Performance. Research is being initiated to determine the simultaneous effect of pertinent fiber properties and combinations of fiber properties on yarn properties and spinning performance to provide guides for obtaining maximum utilization of cottons of varying fiber properties. Work to date has consisted of collection of cottons varying widely in important fiber properties. (S2 1-207).

Large-scale spinning evaluations of the effect of fiber properties and spinning variables on yarn properties and end breakage during spinning have been continued in contract research at Auburn Research Foundation. Results

of spinning tests using the SRRL 720 Spindle Hour Test indicate that yarn twist and spindle speed have a greater effect on end breakage than does short fiber content. Short fiber content had a consistent and adverse effect on end breakage, but this effect was not large except for medium low-twist yarns. Small losses in yarn strength and evenness and lower appearance grades appeared to be associated with high short fiber content. In extending the research to 5,000 spindle hour tests, initial results have verified, in general, findings obtained with the 720 Spindle Hour Test. Three levels of short fiber content and five levels of fiber fineness were evaluated. High short fiber content cotton produced high end breakage, and spindle speed had very little effect on yarn strength until high speeds were reached. Cottons differing appreciably in Micronaire Reading did not have an appreciable effect on end breakage or yarn quality, except at the two extremes. In the case of the short fiber content tests, the relationship between results from the 5,000 versus the 720 Spindle Hour Test were very good; the relationship was not as good for the fiber fineness tests. Generally, yarn twist and spindle speed had greater effects on end breakage than the fiber properties studied. (S2 1-178(C)).

The research on the effect of spotted cottons on product quality and processing efficiency has been completed, except for isolated final evaluations of fabrics. Cottons damaged by Nigrospora oryzae and Rhizopus nigricans, and another sample damaged by Diplodia sp., were evaluated in blends with a white control cotton. The 2.5% blends utilizing Rhizopus- and Nigrospora- damaged fibers exhibited little difference in spinning performance (SRRL Accelerated Spinning Test) compared to the control in spinning a 38/1 yarn. No deleterious effects on spinning efficiency (using the SRRL Accelerated Spinning Test and the 720 Spindle Hour Test) or on yarn strength were noted after "Cavitoma" (high pH, low copper reduction value), Aspergillus flavus, Frost and Clay spotted cottons had been stored for over two years under variable conditions of temperature and humidity. Fourteen months' outdoor weathering tests on fabrics containing various percentages of these four types of spotted cottons showed they were as resistant to outdoor weathering as control cotton fabrics containing no spotted cotton. (S2 1-153).

A fundamental investigation of the causes of warp breakage in the weaving of cotton yarns is being conducted at the Shirley Institute under a P. L. 480 research grant now nearing expiration. The breakage of warp yarns during the weaving of cotton fabrics is the principal deterrent to higher weaving efficiencies. Data from past weaving experiments have been evaluated through the use of a punched card system, and a more detailed study conducted, based on observations of the operations of a slow-motion model loom in conjunction with the insertion of simulated obstructions in selected warp yarns. Evidence has been obtained to indicate that breaks in warp yarns result from abrasion by obstructions and are not due to weak or thin places in the yarns. Results obtained in the studies employing the model loom are being correlated with those obtained in full scale weaving experiments to verify these findings. Increased knowledge of the mechanism of warp breakage and of

means to minimize it is expected to result in improvement in the quality of cotton textiles, and to reduction in costs through increased efficiency in the weaving operation, which represents a large proportion of the cost of producing cotton textiles. (UR-E29-(20)-4).

The relationships between the cohesion of cotton fibers and other physical properties of fibers, rovings and yarns are being investigated in P. L. 480 research at the Juan de la Cierva School of Technical Investigations. The cohesion of cotton fibers affects the roll settings, roll pressures and twists to be used in producing yarns of optimum quality. The main laws governing the minimum twist of cohesion of cotton rovings and yarns in connection with testing conditions (length and tension) and fiber parameters (length and micronaire) and yarn parameters (number of fibers per cross section and twist) have been determined; an improved apparatus for measuring minimum twist of cohesion has been developed; and work is underway to establish the relationship between fiber surface properties and minimum twist of cohesion. It is expected that information developed in this project will permit the relatively rapid and simple measurement of force of cohesion to be used in predicting the spinning efficiency and yarn properties of cottons of differing fiber properties. (UR-E25-(20)-2).

2. Improved Processing Procedures to Obtain Maximum Utilization of Native and Modified Cottons. Research was continued on the development of optimum processing procedures to minimize the detrimental effects of short fibers in cotton spinning performance and product quality. As a means of obtaining cottons differing in fiber length distribution for the research, fiber analyses were performed on a series of California and Arizona cottons harvested and ginned under carefully controlled conditions. In the case of the California samples, short fiber content as well as other length properties varied systematically with harvesting and ginning practices, the short fiber contents ranging from about 4 to 7%. For the Arizona cottons, length was found to be significantly influenced by ginning temperature, number of lint cleaners, and also days. Micronaire Readings were virtually unaffected by harvesting or ginning conditions, as expected. An improved 720 Spindle Hour Spinning Test (whereby constant yarn tension is maintained throughout the increase in spindle speed required) was used in spinning evaluation of 16 selected lots of the Arizona cottons. It was found that decreasing spinning draft and increasing yarn twist can substantially reduce end breakage rates on cottons of inferior fiber length distributions. The influence of draft on end breakage is more pronounced on cottons of inferior length distributions than on superior cottons. Sixteen lots of the California cottons are being processed into rovings to be used for spinning evaluation. (S2 1-179).

Further progress was made in the research to characterize fiber damage in mechanical processing of cotton from opening through carding to provide information needed for developing improved textile machinery and processing methods. Static pressure (hydraulic press) up to 38 tons per square inch caused considerable increase in alkali centrifuge value (ACV) indicating the sensitivity of the test to fiber changes due to pressure. The ACV for

slivers from the granular card indicated greater deteriorative action than the standard card. The picker produced negligible changes in cotton properties when the beater action was that normally used, but with abnormally high beating energy measurable fiber property changes indicated slight deteriorative effects. Fiber breakage and yarn breakage in spinning increased with beats per inch of cotton fiber passing through the picker-beater; also, the yarn strength decreased with beats per inch. The effects of changing card speeds were insignificant, but increases in card production rate increased fiber breakage. Both findings are evidence that mill practices will affect cotton's processing behavior. Temperature and humidity affected fiber breakage and alkali-centrifuge swelling for cotton carded with the Nepotometer. The similar behaviors of cottons of normal drying and extreme drying for these two properties indicate that extreme drying may not produce a potential damage in later processes if normal moisture is restored. (S2 1-185).

In the spinning of cotton yarns, assemblies of fibers are simultaneously drawn out and twisted. The drafting forces exerted during the spinning operation affect the quality of the resulting cotton yarns. Research is being carried out under a P. L. 480 project at the Juan de la Cierva School of Technical Investigations to investigate the effect of various factors in spinning, such as drafting speed, roving twist, apron opening, roll setting, etc., on drafting force in the drafting zones of high draft spinning equipment, and how the drafting forces affect yarn quality. Means have been developed to actually measure the drafting forces in the front and rear drafting zones. Results obtained thus far in the investigations indicate that lowering roving twist and increasing drafting speed increases yarn strength, which means that mills may be able to increase their spinning production with an improvement in yarn strength by this means. The investigation is expected to provide basic information that will be of assistance in developing improved drafting systems, and in making more efficient use of existing systems. (UR-E25-(20)-13).

B. Development of New and Improved Methods and Instruments for Measuring the Physical and Chemical Properties of Cotton

1. Development and Adaptation of Instrumental Techniques for Measuring the Changes Imparted to Cotton by Chemical and Mechanical Processes. Research was initiated to develop reliable test methods to properly evaluate stretch cotton textiles. The tentative techniques developed thus far have been useful in evaluating stretch and recovery of experimental yarns and fabrics. False twisted-stretch yarns have been tested by using a multiple strand specimen consisting of 40 yarns held together at each end with masking tape to determine recovery in addition to breaking load and elongation. A specimen is cycled five times between 0 and 400 g. to measure the work of stretching and the extension of the fifth cycle. To determine permanent set, a specimen is cycled to 400 g. once and allowed to relax for five minutes. The recovery of fabrics has been measured after loads of 0.5 lbs. and 2 lbs. and relaxation times of 5 min., 1 hr., and 24 hrs. Optimum

testing techniques for evaluating stretch cottons will be sought in continued research. (S2 1-212).

Neps, small tangled clumps of fibers that first become visible in the card web from the cotton carding machine, are the cause of serious irregularities or defects in cotton fabrics. The counting of neps in the card web is a necessary quality control measure in the production of fabrics, but it is difficult and time consuming when done by the manual methods usually employed. Research under a P. L. 480 project at the Juan de la Cierva School of Technical Investigations has led to the development of a rapid, automatic scanning device by means of which neps are counted by measurement of irregularities in the transparency of card web samples. This device has been combined with an electronic integrating instrument which will classify the measured neps into four groups according to size, and automatically record the number of neps in each size group in a given sample of card web. Application has been made for a U. S. patent covering the latter development. (UR-E25-(20)-1).

A P. L. 480 research project at the German Research Institute for Textile Industry is being conducted to develop an apparatus for the rapid and automatic counting of neps in cotton card web by means of light reflectance and detection. Progress is being made toward the development of an instrument capable of automatically and continuously measuring and recording neps in the web from a full scale card, based on a prototype developed for use with a laboratory scale card. The development, if successful, is expected to be of great value to cotton processors since it would provide rapid means for following, and perhaps automatically controlling, an important processing variable that affects cotton fabric quality. (UR-E10-(20)-2).

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Effect of Fiber Properties on Processing Efficiency and Product Quality

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Development of New and Improved Methods and Instruments for Measuring the Physical and Chemical Properties of Cotton

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1/ Publication resulting from research under grant of P. L. 480 funds to the foreign institution.

AREA NO. 3 - COTTON - NEW AND IMPROVED TEXTILE MACHINERY

Problem. Cotton is plagued by problems of trash and nonuniformity of fiber length distribution that are not present in synthetic fibers, paper, and other competitive products. Highly efficient methods of cleaning are needed by the cotton textile industry to process satisfactorily the large quantities of machine-harvested and roughly hand-harvested cottons being marketed. Last year more than eleven million bales were harvested by these methods in the United States. Such cottons are difficult to clean with existing textile equipment because of the type of their trash -- largely fine, leaf trash. The development of an integrated system for opening, cleaning, and carding today's cotton can provide substantial improvements in quality and lower costs. The present cotton mill utilizes ten or more processing stages and, compared with other manufacturing systems, an excessive amount of labor. The redesign of existing equipment and the development of radically new types of processing machinery offers an opportunity for major improvements in uniformity and overall quality of textile products, and for savings in manufacturing costs through decreased waste of spinnable fiber, and through reductions in machinery investment, space, and labor.

USDA PROGRAM

The Department has a continuing long-term program involving mechanical engineers, physicists, and cotton technologists engaged in research to design and develop new and improved equipment for processing cotton into higher quality, lower cost consumer products.

Research to develop improved mechanical processing machinery, for opening through carding, is conducted at New Orleans, Louisiana. This work includes the development of experimental machines and pilot scale machines for evaluation under pilot-plant conditions, and subsequent development of plans for scaling up successful units into practical, commercial size equipment. Current research involves the development of a bale-breaker-blender for opening and blending cotton, the improvement of cleaning at the card, and the development of a machine for removing short fibers from cotton. Close cooperation is maintained with cotton textile machinery manufacturers and cotton textile processors in the establishment and dissemination of engineering specifications for the commercialization of new and modified processing equipment. Additional research in this area is being conducted under contract at General Applied Science Laboratories, Inc., Westbury, L. I., N. Y., on the aerodynamic separation of lint cotton into individualized fibers to provide information needed for improving cotton textile processing equipment.

The Federal in-house scientific effort devoted to research in this area totals 15.7 professional man-years. All of this effort is on the

development of improved mechanical processing machinery - opening through carding. The contract research involves an additional 2.0 man-years, in the field of improved mechanical processing machinery-opening through carding.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Improved Mechanical Processing Machinery - Opening Through Carding

1. Equipment for Blending Cottons of Different Fiber Properties to Produce Improved Cotton Textiles. Since cotton varies widely in fiber properties within and between bales, it is essential to blend bales into a homogeneous mass to avoid the production of non-uniform, inferior quality textiles. In order to provide the cotton industry with an urgently needed method for improved blending, research has been directed toward the development of an efficient pilot model bale-breaker blender.

A half-size prototype blender was designed and constructed. Preliminary start-up without cotton pointed to the need for making certain modifications to perfect the hydraulic system used to drive the oscillating head assembly of the machine. This assembly houses the processing cylinders which extract tufts of cotton directly from the bales. By use of a new, special type hydraulic valve to suppress the hydraulic shock previously experienced when the assembly changed directions, continuous operation of the blender at the desired speeds has been made possible. Transverse movement of the bale in combination with the establishment of optimum operational parameters have resulted in the attainment of an exceptionally high production rate. At the high rate of production, however, doffing becomes a major problem and efforts are now being concentrated to achieve adequate doffing. Automation of the feeding operation of the blender was also successfully accomplished. Upon attainment of satisfactory doffing, the machine will be tested and its blending characteristics will be evaluated on a laboratory scale. (S2 1-154).

2. Improved Cotton Carding Machinery for Better Cleaning, Fiber Separation and Orientation. Completed phases of research on modification of the cotton card to improve cleaning have shown that an upper limit exists for trash removal for a set of mechanical conditions, and further improvements in cleaning efficiency and sliver quality will be dependent upon the development of improved methods of feeding the card. More than two stages of the SRRL Fiber Retriever, a mote box modification for the card, were found to be superfluous for removing trash equivalent to the theoretical trash ceiling. Final evaluation at the Southern Division showed that a better cotton yarn is produced with the Retriever. There is a significant improvement in strength; and uniformity and appearance are slightly improved. A card equipped with the Fiber Retriever produces a sliver with about one-third less trash than conventionally equipped cards. The trash removed contains a higher percentage of motes and fibrous trash which are extracted without stripping off the undeveloped fibers usually associated with these types of

foreign matter. Thus, the undeveloped fibers, as well as the trash, do not go forward into the sliver to subsequently degrade the yarn. Two designs of the Retriever (single stage, and double stage) have been released to industry to meet different mill cleaning requirements. Formal testing on high quality cotton by a commercial mill group showed that the Retriever increased trash removal 44% without increasing the loss of fiber in the waste. This organization is installing Retrievers in its cotton mills.

Basic studies of the carding machine at reduced speeds have revealed phenomena that are contrary to accepted concepts of carding. At these slow speeds, air currents and centrifugal forces are negligible, yet a normal carding process apparently takes place. A single test has indicated that yarn produced at slow speed is stronger and slightly less uniform than yarn at standard card speed, substantiating the fact that carding is a mechanical process. The slow speeds will be utilized for future investigations and observations. Work is also underway on designing a satisfactory device for predrafting the cotton lap before it enters the card feed roll. (S2 1-137; S2 1-215).

3. Machine for Removing Short Fibers from Cotton. Research directed toward the ultimate development of a machine for removing short fibers from cotton has shown that length differentiation of fibers by electrostatic means can be accomplished as a continuous process by two methods. One method had excessively low production with the ability to remove from 30 to 50% of the short fibers, while the other method has a higher production rate and less effective short fiber removal. Both uniform and non-uniform electrostatic fields have been applied. As inability to supply individualized fibers at high rates of production appears to be the limiting factor, rather than the electrostatic separating means, emphasis will be placed on developing improved fiber opening means and new methods of introducing fibers into electrostatic fields. Further in-house research on the fractionator, a purely mechanical device under development for separating fibers, has been discontinued since evaluations have not shown sufficient short fiber removal. (S2 1-164; S2 1-164(Rev.)).

4. Aerodynamic System for Separating Lint Cotton into Individualized Fibers. An analytical investigation of the application of aerodynamic forces for individualizing cotton fibers is being conducted in recently initiated contract research at General Applied Science Laboratories, Inc. Basic information derived in initial phases of the work will be utilized in the construction of a laboratory-scale aerodynamic system for separating masses of fibers into individualized fibers. It is believed that aerodynamics offer much promise for application in the field of opening, cleaning and processing cotton. (S2 1-204(C)).

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Improved Mechanical Processing Machinery - Opening Through Carding

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Improved Mechanical Processing Machinery - Drawing Through Weaving

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AREA NO. 4 - COTTON - IMPROVEMENT OF WASH-WEAR PROPERTIES

Problem. Garments which are wrinkle resistant and suitable for wash-wear use are increasingly important to the consumer. Although much progress has been made toward securing this market for cotton, much additional information is needed to hold and expand cotton's share of this enormous market. According to current industry estimates 1.2 million bales of cotton are used annually which would not have been utilized except for the wash-wear development. Projected estimates indicate that in the future most apparel and almost all household textiles will be given a wash-wear or a minimum-care finish. Research on synthetic fabrics is mainly aimed at this lucrative market and is several times greater than the entire utilization effort on cotton. At the same time chemical firms are reducing their research in the development of cotton wash-wear finishes. Promotional advertising claims on cotton wash-wear products have exceeded the actual achievement, and many problems remain to be solved. Much fundamental information is needed to explain mechanisms of the reaction of cotton with crosslinking agents as a basis for the development of new and better wash-wear finishes and for the improvement of present processing techniques. Much applied information is needed which, while essential to the maximum utilization of cotton, is generally beneficial to all processors and therefore comparatively unattractive financially to individual companies. Areas in which research is needed to improve wash-wear cottons include processing techniques, fabric appearance, durability, and comfort. Fabric appearance involves the ability to dry smoothly, resistance to wrinkling or mussing during wear, resistance to dry, wet, and oil soiling, introduction of durable creases as desired, dimensional stability and elimination of seam pucker. Durability involves tensile and tearing strength and abrasion resistance in the finished fabric as well as resistance to abusive laundering, particularly bleaching and souring. Comfort involves moisture absorption during use, elimination of odor on storage or wearing and, in certain cases, stretchability of fabric.

USDA PROGRAM

The Department has a continuing long-term program involving organic chemists, physical chemists, analytical chemists, physicists, microscopists, chemical engineers, mathematicians, cotton technologists, textile technologists and textile engineers engaged in both basic and applied research on wash-wear finishing and improvement of wash-wear properties of cotton. Informal cooperation is maintained with textile finishers, chemical manufacturers, and textile research institutes in connection with the research.

Basic and exploratory research on wash-wear finishing of cotton is conducted at New Orleans, Louisiana. This research is designed to give a better understanding of the chemical reactions and physical changes taking place in wash-wear finishing and the crosslinking of cotton in general. It also

seeks to correlate the properties of the finished cotton with the chemical structure of the crosslinking agents. Basic studies of the relationship of fiber properties to fabric behavior in wash-wear treatments are also conducted. The results provide a broad and sound foundation for the development of new, practical wash-wear finishes for cotton.

Research on the improvement of smooth drying properties -- the essential features of a wash-wear fabric -- is conducted at New Orleans, Louisiana. Some important phases of current work involve development of new crosslinking treatments and optimum wash-wear fabric structures; combination of chemical and mechanical treatments to improve strength and resilience; and pilot-plant evaluation of promising laboratory finishes. Additional research on improved smooth drying properties is in progress under contract at the Fabric Research Laboratories, Dedham, Massachusetts, on investigation of the relationships between fabric structure and ease-of-care performance; and at North Carolina State College, Raleigh, North Carolina, on the effects of mechanical treatments of fabrics prior to, during and following resin finishing on ease-of-care properties.

Research to develop new and improved processing methods for the treatment of cotton yard goods and garments to impart wash-wear properties is carried out at New Orleans, Louisiana. Processing methods are being investigated for the production of wash-wear cotton stretch fabrics with improved strength, drape, and hand. Methods of crosslinking stretch cotton to stabilize the fabric and make the stretch durable to laundering are undergoing study. Other phases of processing research have included studies of methods of drying and curing in the resin finishing of cotton. Cost estimates for new chemicals and for processing of cotton are made to aid industrial establishment of the research developments.

The Federal in-house scientific effort devoted to research in this area totals 30.1 professional man-years. Of this number 12.6 is devoted to basic and exploratory research on wash-wear, 12.8 to research on improved smooth drying properties, and 4.7 to new and improved processing methods. The contract research involves an additional 1.7 man-years, all of the effort being on improved smooth drying properties.

The following lines of work were terminated during the year: (1) Investigations designed to develop reactive finishing agents for cotton that will provide improved elastic and strength properties by the introduction of cellulose crosslinks of optimum size and structure (under basic and exploratory research on wash-wear); and (2) Development of wash-wear cotton fabrics and garments with durable creases and shape holding properties (under new and improved processing methods).

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Basic and Exploratory Research on Wash-Wear

1. Basic Studies of Recovery from Wrinkling and Creasing. In further investigation of wet and dry crease recovery mechanisms in wash-wear cotton products, certain relationships between types of crosslinking and physical properties have been found but possible causes for the behavior need further exploration. It has been observed that tensile recovery behaviors usually reflect wash-wear behaviors but the relationships are extremely complex. In general, tensile recoveries of resin-treated cotton usually agree with crease recovery; however, discrepancies are found, such as dry recoveries without wet recoveries, but having significant tensile recoveries. The strain recoveries observed for cotton and rayon at standard and wet conditions indicate difficulties in selecting a single condition for characterizing resin treatment on different materials. Low temperature, long-time curing of fabric with methylol-amide crosslinking agent gave a small but real improvement in strength/crease-recovery relationships. Use of additives to maintain swelling in dry-cure crosslinking reduces the number of crosslinks formed but the reduction is not sufficient to explain the loss in dry-crease recovery. Microscopical techniques have been developed which make it possible to determine if crosslinking has occurred throughout the fiber or on the periphery only, and whether crosslinking has been induced under swollen or unswollen conditions. Effort will be concentrated on the relationships between physical behaviors, optical observation of changes, and the chemical reactions considered to have occurred. (S2 1-189).

Research was initiated to investigate the relationship of fiber properties to fabric behavior in wash-wear treatments. Special effort will be made to elucidate the exact role played by various fiber properties in fabric wrinkle recovery, tensile strength, elongation, and tearing strength. In initial experiments, good correlations between wrinkle recovery and a special measure of elastic recovery for crosslinked fabrics of a wide range of properties have been obtained, showing that elastic recovery plays a vital role in wash-wear behavior. However, other properties such as fabric modulus must be considered in the relationship. In studies of DMEU resin treatment of fabrics, it was also found that dynamic flexural rigidity is less affected by resin treatment and by tension during treatment than is tensile modulus. This is the result of reduction in fabric thickness on resin treatment, especially when tension is applied. A small but consistent increase in density was found for add-on of MMM resin up to 2%. Higher add-on decreases density. This may indicate that about 2% add-on of resin goes into void spaces of the fiber without increasing the volume. Comparison of ordinary practical measurements of "immediate" elastic recovery with a method of estimating true zero time recovery indicated that the former procedures may give misleading results in certain applications. The effect of humidity on wrinkle recovery and elastic recovery of various types of crosslinked fabrics will be investigated. (S2 1-198).

An investigation of the effects of time and environmental conditions on the rate of wrinkle recovery of wash-wear cotton textiles has been initiated. It is likely that both rate of wrinkle recovery and ultimate crease recovery affect wash-wear properties. Preliminary experiments on rate determination (confined to fabrics finished with two crosslinking agents) indicate an initial rapid recovery from creasing to about 90% of total recovery in less than 30 seconds, followed by a greatly decreased rate of recovery as time increases. Research will be continued along the present lines to develop a suitable method for determining rate of wrinkle recovery. Cotton treated with other finishing agents will be investigated. (S2 1-203)

2. Variations in Physical Properties of Cotton Induced by Crosslinks of Different Chemical Structure. Contract research at the Lowell Technological Institute Research Foundation on development of reactive finishing agents with optimum size and structure for wash-wear cotton was continued and completed. Three new series of crosslinking agents, including agents with three different reactive groups and two different inert groups to govern the length of cellulose crosslinks formed, were prepared. The agents [dicarbamates of polyethylene glycols; bis(N-methyl-carbamates) of polyethylene glycols; and alkane bis(hydroxyethylsulfones), the first two series in the form of their methyl derivatives] were applied to cotton fabrics and evaluated. Results show that significant improvement in the mechanical properties of wash-wear cotton cannot be achieved by merely lengthening the crosslinks, even to a distance of 30 atoms. Since modification of the size of crosslink is ineffective, other approaches will need to be followed in research aimed at the preparation of stronger wash-wear cotton. (S2 1-148(C)).

A promising new wash-wear finish, based on the use of tetrakis(N-methylolcarbamoylethyl)ethylenediamine, has been found in research on the development of new types of N-methylol finishes to produce high quality, durable wash-wear cotton fabrics. The finish appears to be equivalent to the previously developed tris(N-methylolcarbamoylethyl)amine (TMCEA) finish. The susceptibility to chlorine damage of some dimethylol ethyl carbamate (DMEC) finishes has been traced to use of sodium carbonate in the methylolation procedure. The commercialization possibilities of both the TMCEA and DMEC finishes continue to be good.

Wash-wear cottons with promising properties have also been prepared using N-methylol compounds containing both tertiary amine and carboxylic acid groups. Application to cotton is simplified by the presence of both these groups simultaneously because they form an inner salt and allow the reaction to take place under mild catalysis. During washing and bleaching of the fabric the alkali salt of the acid group is formed, thereby providing added protection from scorch damage. In another phase of work, it has been demonstrated that hydrazide-formaldehyde wash-wear finishes are based on nitrogenous and/or formaldehyde crosslinks. When a nitrogenous crosslink provides the wash-wear properties, hypochlorite removes the wrinkle resistance properties but increases the tearing strength of the fabric. A new

triazone, 2-methoxyethyl, has been found to offer several advantages over the triazones in commercial use for wash-wear finishing, but its cost could eliminate or reduce its use. (S2 1-177).

B. Improved Smooth-Drying Properties

1. Controlled Reaction of Formaldehyde With Cotton. In further work on development of wash-wear cotton fabrics by controlled reaction with formaldehyde, a gaseous formaldehyde treatment for cotton has been found which requires only a short reaction time (less than 8 minutes) to produce good wrinkle resistance and wash-wear properties. It has also been established that magnesium bromide and iodide are more effective catalysts than those previously used for pad-dry-cure formaldehyde treatments. Dry and wet crease recovery and other physical properties of cotton crosslinked in the wet state by formaldehyde solutions are affected by almost any change in reagent composition, but water concentration in the treating bath does not influence the relation of dry crease recovery to bound formaldehyde. The effects are more complicated than believed previously and seem related to crosslink distribution within the fiber. Appreciation of the specific effects of each of the many factors affecting the properties of crosslinked cotton fabric should lead to additional modifications of industrial finishing treatments that will give improved wash-wear fabrics. (S2 1-166).

2. Development of New Treatments to Improve Strength and Resilience of Wash-Wear Cottons. Research was continued on the application of crosslinking treatments to various chemically modified cottons as a possible means of improving wrinkle resistance, strength and other desirable properties for wash-wear cotton textiles. Various carboxyl-substituted chemically modified cottons treated with nitrogenous crosslinking agents have been found to exhibit high resistance to chlorine damage in the scorch test due to the protective buffering effect of the salt form of the carboxyl groups. Of the modified cottons studied, hydroxyethylated cotton appears to be the most amenable to the production of good wet and dry wrinkle resistance by gaseous formaldehyde treatment.

Some relations between distribution of crosslinks and crease recovery properties in crosslinked cotton have been shown in studies of the treatment with DMEU of cotton acetylated to various degrees, including partially deacetylated cotton. Interlamellae (between growth layer) crosslinks are necessary for dry crease recovery. Intermicrofibril crosslinks accompany interlamellae crosslinking, and as a result the fabrics have wet and dry crease recovery. Only intermicrofibril crosslinks are needed for wet crease recovery development. Proper use of this fundamental information should provide better wash-wear cotton fabrics.

Cotton fabrics possessing stretch properties markedly greater than those of slack mercerized cotton have been produced by first chemically modifying cotton under tension with groups which impart alkali-swellability and then treating the modified cotton with alkali solution. These products can be

crosslinked to give wash-wear fabrics retaining stretch. Load-elongation curves have indicated that crosslinking slack mercerized-carboxymethylated cotton decreases elongation only slightly at any given load. Strength and ultimate elongation are reduced, however, as is the normal consequence of crosslinkage. (S2 1-171).

Work was initiated under contract at North Carolina State College to determine the effects of mechanical treatment of cotton fabrics prior to, during, and following resin finishing on the ease-of-care properties of the fabrics. Grey fabrics (80 x 80 print, broadcloth, and three-leaf twill) were pretreated by singeing, desizing, scouring, and bleaching processes comparable to commercial practices. The three basic fabrics were then mercerized under three tension conditions (minimum tension, warp and filling; maximum tension, warp and filling; maximum tension, warp only). The mercerized fabrics, and suitable controls, were wet out and dried on a tenter frame using the same tension conditions as employed for the mercerizations. Portions of the resultant fabrics were subjected to mechanical compressive shrinkage to reduce residual shrinkage to 1 percent or less. It is planned to employ ethyl carbamate, APO and DMEU for resin treatment of the various fabrics. Various ease-of-care properties of the final fabrics will be evaluated. (S2 1-183(C)).

3. Development of Optimum Wash-Wear Fabric Structures. In research on development of optimal structures for cotton fabrics for wash-wear products, further testing of the experimental fabrics designed to determine the effect of type twist on properties of DMEU-treated fabrics showed that the fabric with all Z-twist yarns in general exhibited higher breaking and tearing strengths than the fabric combinations containing S-twist yarns. In another completed phase of the research it was found that yarn uniformity and uniformity of tensions in weaving do not produce significant effects on wash-wear ratings of cotton fabrics after DMEU resin finishing. Fabric strengths were slightly higher in the fabrics made with uniform yarns, as compared to those prepared from non-uniform yarns.

Present in-house work involves investigations of methods for introducing additional yarn length between cross-yarn contacts within fabrics to relieve bending stresses without altering the finished construction. Two approaches are currently being used: (1) the additional yarn length is introduced into the fabric by mechanical means during weaving, and (2) chemical means are employed after the fabric is woven. A completed series of tests using the mechanical approach show that surprisingly good wash-wear properties can be achieved by treating the fabrics with an extremely low resin concentration (2% DMEU solution). However, a control fabric prepared under normal weaving conditions and processed along with the test fabrics gave similar results. The cause for this unusual behavior of the control sample is now being sought. Tests on a series of experimental fabrics prepared by the chemical approach are underway. (S2 1-163).

Contract research at Fabric Research Laboratories on relationships between ease-of-care performance and the geometry (structure) of cotton fabrics has indicated that, at the levels of resin add-on conventionally employed in wash-wear finishing, the effects of fabric structure on wash-wear behavior are not very pronounced. Flexural rigidity of bleached fabrics and bleached, DMEU-treated fabrics changed only slightly, if any, with increase in humidity. Bleached, APO-treated fabrics became stiffer while mercerized, APO- and mercerized, DMEU-treated ones became less stiff with increase in humidity. The increased stiffness appears to have no adverse effect on wrinkle recovery. All of the treated fabrics were found to recover rapidly with high humidity, and the immediate crease recovery (within one minute) was almost as great as the delayed recovery (five minutes). Except for the APO treatments, the delayed recovery angle is no greater at high humidity than at 65% relative humidity. The immediate recovery was always higher at high humidity. When wash-wear ratings of the fabrics were comparable, differences associated with structure were found in flexural rigidity and crease recovery. These differences were found between heavy- and light-weight fabrics, and also between extremes in structures for fabrics of comparable weight. Fabric structure also affected resin add-on level. Upon completion of analysis of data, recommendations will be made as to fabric structures which improve wash-wear behaviors. (S2 1-170(C)).

C. New and Improved Processing Methods

1. Development of Durably Creased, Shape-Holding Garments. The research to develop wash-wear cotton fabrics and garments with durable creases and shape-holding properties was completed. In recent work it was found that durable creases can be imparted to cotton, crease resistant cotton fabrics, and certain other chemically modified cottons by treatment with nitrogen dioxide. Durable creases have also been produced in cotton fabrics by wet-fixing methylated methylolmelamine in the fabric using aluminum chloride catalyst, washing, then impregnating with zinc nitrate, shaping with a hand iron, and completing the "cure" in an oven. The fabric may be stored for a period of time after the zinc nitrate impregnation step before imparting the creases. Urea-formaldehyde and dimethylolethyleneurea may be similarly used in this process. The fabrics produced by this wet cure technique have moderate to high wet and dry wrinkle resistance, and high moisture regain, in addition to the durable creases. The research also showed that wash-wear appearances of crosslinked cotton stretch fabrics are greatly superior to those of the slack mercerized controls after washing and line-, drip-, or tumble-drying; and there is no loss of stretch properties. (S2 1-162).

2. Wash-Wear Cotton Stretch Fabrics With Improved Strength, Drape and Hand. Investigations of finishing treatments to produce wash-wear cotton stretch fabrics with improved strength, drape and hand have been initiated. Samples of 72 specially woven fabrics have been obtained to determine the effects of yarn and fabric structure on strength, drape, and hand of wash-wear cotton fabrics. An instrument has been designed and constructed for restretching fabrics before, during and after slack mercerizing, and also

before crosslinking, to determine optimum conditions of preparation. The instrument is currently being tested. In crosslinking with alkali-catalyzed agents, it has been discovered that several processing steps may be eliminated by adding the agent before all the alkali is removed in the shrinkage step. Production of wash-wear stretch cottons by this technique should be economically attractive. (S2 1-211).

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AREA NO. 5 - COTTON PRODUCTS WITH SPECIAL PROPERTIES

Problem. In many uses where special properties are of paramount importance, cotton is being replaced by synthetic materials. To improve its position in the textile market, which has declined from 79.5% of mill consumption of all textile fibers in 1939 to an estimated 60% in 1962, new applications must be explored and improved products developed to meet the competition of synthetic fibers. Cottons having high recoverable stretch, durable loft, light-weight bulk, pleasing textures, warmth and other highly desirable properties are needed to enable cotton to compete successfully with synthetic fibers in the rapidly expanding market for stretch and bulked type fabrics. Fabrics designed to achieve increased resistance to tearing and abrasion, flex life and other strength properties are needed to improve the wear life of cotton textiles for apparel, household and industrial uses. Cotton fabrics must be designed to withstand better the elements of weather and finishes developed that will provide greater protection from solar radiation, microorganisms, acids and fire, and that will resist color change. Cotton fabrics with improved heat and scorch resistance are needed in the commercial laundry industry and for home ironing board covers. Additional basic information must be developed to improve cotton's resistance to water and oil-borne soils, and to dry soiling. Resistance to soiling ranks fifth in importance among the 40 end-use qualities for textiles. Cheaper and durable flame retardant finishes for cotton, specially for outdoor use, are needed. Numerous consumer preference surveys have shown that a great potential demand exists for cotton material that will be more lustrous without sacrifice of functional properties. Cotton textiles with multipurpose finishes are also needed, particularly those where several desirable end-use properties can be introduced in a single process.

USDA PROGRAM

The Department has a continuing long-term program involving organic chemists, analytical chemists, physical chemists, physicists, microscopists, chemical engineers, cotton technologists, textile technologists and textile engineers engaged in both basic and applied research to develop new or improved cotton products possessing special properties to meet the competition of synthetic fibers and other synthetic materials in various end uses. Informal cooperation is maintained with textile finishers, chemical manufacturers, and textile research institutes in connection with the research.

Research is carried out at New Orleans, Louisiana, in cooperation with the Foundation for Cotton Research and Education (affiliated with the National Cotton Council of America) and the Canvas Products Association International, to develop cotton fabrics with improved resistance to outdoor weathering. This research includes discovery of new and more effective fungicides, and sunlight and microbial resistant pigments for cotton textiles; and

development of improved formulations, equipment and procedures for producing weather resistant cotton textiles. Additional research is being conducted under contract at Texas Woman's University, Denton, Texas, on development of weather-resistant, water-repellent finishes for cotton.

Research to develop new fluorochemical finishes for oil- and water-repellency and other reactive and additive finishes is conducted at New Orleans, Louisiana, to improve cotton's soil resistance. Additional research is being performed under contract at the Harris Research Laboratories, Inc., Washington, D. C., to provide fundamental information on the mechanism of the soiling of cotton by water- and oil-borne soils. This research includes a determination of the effects of surface change, oil and water repellency, hardness and thermoplasticity (of coated fabrics) and their relationship to the ease of soiling and soil removal from cotton.

Research on flame resistant cotton textiles is performed at New Orleans, Louisiana. Recent emphasis has been on the development of treatments to impart flame resistance to cotton while at the same time imparting other desired textile properties.

Investigations of methods for imparting durable luster and related appearance characteristics to cotton textiles are carried out at New Orleans, Louisiana.

Research to improve cotton's bulk, elasticity and resilience through resin treatment, chemical modification, slack mercerization and other type swelling treatments, of fibers, yarns and fabrics is conducted at New Orleans, Louisiana. The research on fibers is aimed primarily at the development, by chemical or mechanical means or both, of more resilient and cohesive cotton batts for use in mattresses and other padding applications in the furniture and automobile industries. The cotton batting research is conducted cooperatively with the National Cotton Batting Institute, Textile Waste Association, National Cottonseed Products Association and the Foundation for Cotton Research and Education (affiliated with the National Cotton Council of America). Work on yarns is intended to produce bulky, elastic yarns suitable for weaving or knitting into fabrics with improved stretch and bulk characteristics. Investigation of a slack mercerization process, with and without subsequent resin treatment, is being carried out to achieve stretch cotton fabrics for industrial, household and apparel uses. Additional research on stretch and bulked cotton products is being carried out under contract at North Carolina State College, Raleigh, N. C., on evaluation of stretch-type cotton yarns in knit wear; and at Clemson Agricultural College, Clemson, S. C., on development of cotton knit fabric having increased bulk, warmth, and dimensional stability.

Research on the effect of yarn and fabric construction on the physical properties of chemically treated cotton fabrics is another phase of work at New Orleans, Louisiana. Engineering studies are in progress to determine the feasibility and practicability of chemical and resin treatment of

roving by continuous processing as an intermediate step in the mechanical processing of cotton. Chemical, resin, or combination treatment of roving could provide new and improved properties useful in such applications as heavy woven products, knitted paddings, tightly twisted or plied yarns and thermoplastic yarns. Additional research is being carried out under contract at the Philadelphia College of Textiles and Science, Philadelphia, Pennsylvania, to develop yarns by mechanical, chemical and physicochemical methods to produce cotton crepe fabrics which will compete with those made from synthetic fibers.

The Federal in-house scientific effort devoted to research in this area totals 32.6 professional man-years. Of this total, 4.9 is devoted to weather resistant cotton fabrics, 2.8 to soil resistant cotton textiles, 4.0 to flame resistant cotton textiles, 3.1 to cotton textiles with improved luster, 15.7 to stretch and bulked cotton products, and 2.1 to effect of yarn and fabric construction on the physical properties of chemically treated cotton fabrics. The contract research involves an additional 3.5 man-years, 0.5 being on weather resistant cotton fabrics, 0.9 on soil resistant cotton textiles, 1.6 on stretch and bulked cotton products, and 0.5 on the effect of yarn and fabric construction on the physical properties of chemically treated cotton fabrics.

The following line of work was terminated during the year: (1) Development of improved winter-weight cotton fabrics on the woolen processing system (under stretch and bulked cotton products).

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Weather Resistant Cotton Fabrics

1. Improved Fungicides, and Sunlight and Microbial Resistant Pigments for Cotton Textiles; and Improved Formulations, Equipment, and Procedures for Producing Outdoor Cotton Textiles. Cooperative research on outdoor fabrics with the Canvas Products Association International and the Foundation for Cotton Research and Education has resulted in the finding that a number of inorganic salts (fungicides) are soluble in zirconyl acetate solutions, probably through complex formation. Cotton fabrics treated with the solubilized fungicides are resistant to algae as well as mildew and rot. Many conventional fungicides are effective for the prevention of mildew and rot but do not prevent the growth of algae. One of the new treatments (copper zirconyl boro-acetate) has attracted keen industrial interest.

New water-soluble fungicides based on zirconyl ammonium carbonate and copper or mercury compounds have also been developed for weather- and rot-resistant cotton finishing. The copper borate-zirconyl ammonium carbonate treatment shows particular promise. The new type process appears to offer advantages over the zirconyl acetate technique in both cost and effectiveness and will be investigated further.

Outdoor exposure and soil burial testing of several metal tellurides and sulfotellurides indicate that these materials may be useful as fabric fungicides. Another promising development is a one-bath system for chromium oxide mineral dyeing. Fabrics treated by this process compare favorably in appearance with commercial samples prepared by the conventional two-bath process. (S2 1-156).

B. Soil Resistant Cotton Textiles

1. Fluorochemical and Other Soil Resistant Finishes for Cotton. Several approaches to develop durable and efficient oil and water repellent finishes, and soil resistant finishes for cotton products have continued. Improved finishes of this type could significantly strengthen the competitive position of cotton in domestic, military and industrial applications.

Further work was done on APO-based treatments for water and oil repellency. Dynamic and static water repellency tests on fabrics treated with APO-stearic acid have indicated that this water repellency treatment has promise. Fabric treated with APO-perfluorooctanoic acid gave very high oil repellency ratings, but improvement in the pad bath stability for this and the APO-stearic acid treatment is needed. Recent studies indicate that certain catalysts based on BF_3 may be compatible with the system and may give the desired stability. Several synthesized derivatives of perfluorooctanoic acid have been evaluated as cellulose reactants with some success.

The reaction of pyridines and of triphenyl phosphine with certain cyclic perfluoroolefins was studied as a basis for producing compounds for evaluation as oil and water repellent or fire retardant finishing agents for cotton. Pyridinium betaines are formed in the first reaction, and presumably phosphonium betaines in the latter. Attempts to transform these pyridinium betaines into cellulose-reactive compounds have failed thus far. During the preparation of one of the betaines, however, an intermediate which should be quite reactive with cotton under anhydrous conditions was isolated. Research is also in progress to prepare fluorinated alpha-chloromethyl ethers, a promising class of cellulose-reactive compounds, using modifications of older procedures. (S2 1-180).

In further investigation of various finishes with respect to soiling and soil removal, formaldehyde crosslinked (Form D) fabrics with cationic, anionic and non-ionic surfaces were found to soil more in aqueous carbon black dispersions at pH 2 and pH 10. The fabrics soiled less at pH 7, and the soil was easier to remove when laundered. Basic oily carbon black dispersions soiled the fabrics more readily than did the acidic oily dispersions; ease of soil removal was not affected significantly. Aminized cotton (anion exchange cotton) was shown to be a scavenger for soil during laundering as it prevented soil redeposition. Phosphonomethylated cotton (cation exchange cotton), which resists soils, did not act similarly. Selected water repellent finishes improved cotton fabrics' resistance to aqueous and oily carbon black soils. Aqueous soil was readily removed from the water

repellent finished samples when laundered, but oily soil was held very tenaciously. An APO-fluorocarbon oil repellent finish did not improve soil resistance, but APO-carboxymethyl cellulose and DMEU-carboxymethyl cellulose treated cotton fabrics resisted repeated soiling better than did untreated cotton. The latter finishes were more effective in aiding removal of oily soil than aqueous soil.

Modified polyethylene polymers containing reactive carboxyl groups and acetate side chains were applied to cotton fabric and evaluated for wet soiling and ease of soil removal. The finishes did not resist aqueous or oily soils, nor significantly affect ease of soil removal. Similar results were obtained with chemical treatments based on a uron resin, glycerol-dichlorohydrin, formaldehyde (pad-dry cure method), stearyl chloride, and thionyl chloride. Other studies will be made to evaluate new finishes and chemical treatments for soil resistance. (S2 1-191).

Fundamental investigations of the effects of specific type finishes on soiling of and soil removal from cotton are continuing under contract at Harris Research Laboratories, Inc. so that the overall mechanism of soiling and soil removal will be better understood. The basic information developed should aid in designing a superior soil resistant finish for cotton.

The contract research has shown that thermoplasticity, adhesiveness, and surface energy of the fabric-water interface at various temperatures affect the way fabrics soil. Soft polymer finishes and those with high energy finish-water interfaces have high levels of soiling. Estimations have been made of the relative surface energies of several cotton finishing agents and the relation of these values to resistance to soiling and ease of soil removal from corresponding finished fabrics. In air, the order of increasing surface energy of smooth films of the finishing agents was perfluoroacrylate<silicone<hydrocarbon acrylate. The same order of resistance to wetting was found for the corresponding finished fabrics. In water, the order was hydrocarbon acrylate<perfluoroacrylate<silicone, and the corresponding fabrics had the same order with respect to resistance to wetting by organic liquids, with unfinished cotton showing the greatest resistance. In the presence of a detergent solution, a similar order of resistance to soil removal was observed. Oil-soiled fabrics ranked in the same order after laundering. Streaming potential measurements on chemically modified fabrics will be made and related to the tendency of the fabrics to become soiled. (S2 1-175(C)).

C. Flame Resistant Cotton Textiles

1. Treatments to Impart Flame Resistance and Improved Textile Properties to Cotton. Continued progress was made in research to develop improved treatments for imparting flame resistance and other desired textile properties to cotton.

Several processes for imparting flame resistance to cotton were developed on a laboratory scale and successfully translated to the pilot plant. Precondensates of THPC with urea, ethyl carbamate or tris(2-carbamoylethyl) amine are applied to the fabric, then fixed with ammonia. The latter formulation shows particular promise, giving fabrics with good flame resistance which is durable to laundering and resistant to damage by chlorine bleaching. Further research will be conducted to improve the efficiency of the ammonia fixation.

Two new products, the formaldehyde derivatives of tris(2-carbamoylethyl) phosphine (TCEP) and tris(2-carbamoylethyl)phosphine oxide (TCPO), have been discovered which simultaneously impart flame and wrinkle resistance to cotton. They are potentially inexpensive chemicals. Work will be continued to establish optimum conditions for their use.

In extending the earlier work on simultaneously applying reactive dyes and resin forming monomers to cotton, it was found that difunctional and hexafunctional aziridinyl-phosphorus compounds are not as effective as trifunctional APO in attaching dyes and imparting crease resistance to cotton textiles. (S2 1-190).

D. Cotton Textiles With Improved Luster

1. Processes for Imparting Durable Luster and Related Appearance Characteristics to Cotton Textiles. New research was initiated to explore chemical and mechanical treatments that might impart durable luster and related appearance characteristics to cotton yarns and fabrics. Fabrics possessing these characteristics are in great demand for many types of clothing and household items.

Initial work has shown that the luster imparted to yarns by mercerization under tension is highly durable to repeated laundering. Gloss or luster applied to fabrics by calendering or schreinering was not durable to laundering; however, subsequent crosslinking treatment made the gloss fairly durable. A highly lustrous fabric was woven from yarn which had been kier-boiled, gassed, and mercerized to 3% stretch. The luster was durable to dyeing with direct dyes. It has been found that the luster in the fabric woven from tension-mercerized yarn is fairly durable to slack remercerization. This has made it possible to prepare a fabric having moderate stretch as well as luster. The luster obtained by weaving fabric from tension-mercerized yarn was increased by going from a broadcloth to a sateen weave, showing that the use of pretreated yarns in various constructions is a promising field for industrial development. One commercial firm has expressed interest in the use of tension mercerization on cotton yarn to obtain luster. Future research will be directed toward physical and chemical treatments to increase the luster obtained on fabric constructions previously woven from pretreated yarn. (S2 1-194).

E. Stretch and Bulked Cotton Products

1. New and Improved Processes for Production of Stretchable Cotton Yarns and Fabrics Using Chemical and Mechanical Treatments. Highly stretchable textured cotton yarns have been made on false twisting equipment at delivery speeds up to 20 yards per minute, using a 30-inch long experimental heater and relatively low heater temperatures (425° F.). The DMEU resin was applied to the yarn continuously during the false twisting operation. Since the delivery speed is limited primarily to the time required for drying and curing the resin-treated yarn, increased rates of production can be obtained by using higher heater temperatures, by increasing the length of the heater, or by employing a faster curing resin. Investigations to determine the effect of yarn structure for 24/2 false twisted stretch yarns on yarn properties such as strength, elongation, recovery, and work of stretching have been completed, and optimum structure for this type stretch yarn has been established. Research to determine the suitability of different types of crosslinking resins for producing the false twisted stretch yarns is now underway. Preliminary results showed that the amount of stretch increased and strength decreased with increases in the amount of resin. DMEU resin produced yarns having more stretch than those made using APO or DMEC resin. Preliminary twist studies have shown that the uniformity of stretch-type cotton yarns produced by resin-treating highly twisted plied yarns in package form, curing with dielectric heat, and then backtwisting can be considerably improved by increasing the amounts of ply and backtwist. (S2 1-193; S2 1-193 (Rev.)).

Stretch yarns having a significant degree of stretch and elongation at break have also been obtained by modifying highly twisted 14/2 cotton yarns by grafting polyacrylonitrile or by cyanoethylation, then backtwisting. Samples containing from about 20 to 30% add-on of the grafted polymer possessed some of the physical characteristics desired for stretch yarns. Cyanoethylated yarns with about 12% add-on exhibited similar properties, but had superior appearance. (S2 1-213).

Evaluation of stretch-type cotton yarns in knit-wear will be made in contract research recently initiated at North Carolina State College. The objective of the work is to develop processing techniques for knitting the stretch yarns into various structures of circular knit fabrics, and to correlate the physical properties of the knit fabrics with performance under actual wearing and service conditions of garments made from these fabrics. (S2 1-197(C)(Rev.)).

Contract research at Lowell Technological Institute Research Foundation on winter-weight cotton fabrics from yarns made on the woolen system has been completed. Resin-treated fabrics made from yarns spun on the woolen system will be limited to very heavy materials due to the limited fineness which can be spun on this system and their low strengths caused by the use of unparalleled cotton fibers. These fabrics had good dimensional stability to drycleaning and laundering, but the resins lowered their

tearing strength significantly. Repeated drycleaning resulted in heavy pilling of the fabric surface and lowered the insulating properties of the fabrics. (S2 1-140(C)).

2. New and Improved Processes for Production of Stretchable Cotton Textiles Using Slack Mercerization and other Type Swelling Treatments. Methods of producing stretch cotton fabrics by slack mercerization for various end uses have been demonstrated in other research. The textile industry has been provided with valuable information on processing techniques to achieve 1-way and, in some cases, 2-way stretch; the effect of yarn and fabric structure on stretch properties; physical properties and recovery characteristics of the stretch fabrics; and equipment and processing cost estimates. At least nine finishers in this country are in commercial production of slack mercerized stretch fabrics, and several others are preparing samples on a pilot-plant scale. Most of the production is going into apparel, but upholstery, slip cover material, and coated fabrics are also being produced. Interest is also being shown in slack mercerized yarn for stretch knit goods. Two manufacturers of men's hose are producing experimental all-cotton stretch hose by slack mercerizing a very loosely knit hose.

Basic information is being obtained on the stretch properties that can be imparted to cotton by shrinkage in selected swelling-type reagents. Studies are in progress to determine the relationship of yarn shrinkage, tension developed, and the swelling of yarn in various swelling solutions. In sodium hydroxide solutions, maximum shrinkage and tension occurred at 32% concentration, while maximum swelling occurred at 23%. In potassium hydroxide solutions maximum shrinkage, tension, and swelling occurred at 32%, although at 23% swelling was nearly as great. This information should be of value in explaining the mechanisms that occur in slack mercerization to produce stretch yarns. (S2 1-187; S2 1-213).

3. Resilient Cotton Batts from Low Cost Cotton. In further research to improve the bulk resilience and cohesion of cotton batting -- conducted in cooperation with the National Cotton Batting Institute, The Textile Waste Association, The Foundation for Cotton Research and Education, and the National Cottonseed Products Association -- resin and latex formulations have been developed which contribute improved performance, for example under high relative humidity conditions. A statistically designed series of experiments has delineated the contribution of the latex and the resin to the composite batt structure in terms of performance, and the effects of important processing variables on the properties of the batt. Results from initial experiments to establish the effects of a significant amount of the fibers arrayed in other than horizontal positions were not as good as hoped for. Work will continue on screening of resins/latexes and on orientation of the fibers in the batt in an effort to improve the product.

Two commercial plants have installed pilot lines for the production of the new cotton batting, and eight additional companies are contemplating the

installation of pilot lines. Tests on auto seat cushions at two major automobile manufacturer have shown that the cotton batting at the present stage of development substantially meets their requirements for cushioning materials. Sample mattresses made with the batting are currently undergoing evaluation. These developments should make it possible for cotton batting to better meet the serious competition from polyurethane foams and foam rubber in padding applications and in mattresses. (S2 1-181; S2 1-181(Rev.)).

F. Effect of Yarn and Fabric Construction on the Physical Properties of Chemically Treated Cotton Fabrics

1. Effect of Fabric Structure on Chemically Treated Fabrics. Research was continued to determine the feasibility and practicality of chemical, resin or combination treatment of cotton roving. Treatment at this stage of cotton processing could provide new and improved properties useful in such applications as heavy woven products, knitted paddings, tightly twisted or plied yarns, and thermoplastic yarns.

In further experimentation with the new type, single cell apparatus developed for chemical treatment of roving, basic operating conditions necessary to obtain rapid, reproducible and uniform wetting of the roving packages at selected pick-ups have been established. A package of roving can be wetted in 1 to 2 minutes. Wetting uniformity within the package can be obtained within 1-2% at wet add-ons ranging from 80 to 150%. Such control should make possible the accurate chemical and/or resin treatment of roving.

Exploratory dielectric curing experiments were carried out with packages of roving treated in the single cell apparatus with APO and urea-formaldehyde resins. Operational difficulties were encountered in the former trials, but the packages treated with urea-formaldehyde were satisfactorily processed and cured. The yarn spun from the roving is undergoing evaluation. Experience gained in the exploratory work should be invaluable in the future operation of this equipment to produce products possessing desired properties. (S2 1-184).

Contract research at the Philadelphia College of Textiles and Science on methods of production of acceptable cotton crepe fabrics was continued. Cotton crepe fabric made from yarns produced by plying S- and Z-twist single yarns together in S- and Z-directions and using alternate pairs in the warp and filling appears interesting and would be less expensive to produce than the yarns obtained by raveling resin-treated knitted fabrics as previously reported. The contractor is carrying out experimental weavings of small yardages of fabric from these new crepe-type yarns. Based on the physical properties of these fabrics, 50 yards of the most suitable crepe fabric will be produced for evaluation. Results to date have indicated that the cotton crepe fabrics produced will not compete with rayon crepes in the same weights. However, it appears that the methods and yarns developed may allow the economic production of 100% cotton fabrics with

novel surface textures, which have minimum-care properties without the use of resin finishes. (S2 1-157(C)).

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AREA NO. 6 - COTTONSEED PROCESSING AND PRODUCTS

Problem. Cottonseed products, currently approximately two billion pounds of oil and 2.7 million tons of meal derived from the annual domestic production of cottonseed, face increasing competition for markets. For its chief market, edible products, cottonseed oil must compete with other vegetable oils and animal fats. The nation's capacity for producing these oils and fats is so great that supplies can be expected to exceed both domestic and foreign demand for some time to come. Cottonseed meal, used chiefly as a protein supplement in feeding ruminant animals, faces serious competition from synthetic urea and other supplements. Improvements in the quality and utility of cottonseed oil are needed to retain present and open new markets for the currently large and possibly greater future production.

As an illustration, there is a discrimination in the markets against 25% to 50% of the current production of cottonseed oil due to the presence of reddish colors that are not removed by present commercial refining, bleaching and deodorizing methods. It is essential that information be developed on the chemistry of the pigments responsible for the off-colors, and that practical means be found to eliminate them and thus upgrade the oils, particularly for use in margarine and shortening. Additional information is needed urgently on the chemical, physical, and biochemical properties of cyclopropene fatty acids in cottonseed and means of converting them, if found necessary, into physiologically inert forms. New types of modified fats, such as polyester and polymeric fats, need to be developed from cottonseed oil for applications in the fields of edible and inedible coatings, waxes, resins, plasticizers, and lubricants. Cocoa butter-like fats and other confectionery fats derived from cottonseed oil could also provide new markets for large quantities of oil. Fundamental information is needed on hydrogenation to permit production of improved plastic fats. Other areas in which markets for cottonseed oil need to be developed through research include fat emulsions for intravenous feeding, edible emulsifiers, and fatty acid amides and derivatives for use as plasticizers, plastic foams and other industrial products. Improvement in the quality and nutritive value of cottonseed meal is needed so that it can better compete with other protein feed supplements. Additional information is needed on the physiologically active constituents of the meal responsible for egg abnormalities, swine mortalities and growth abnormalities of young animals that limit cottonseed meal's usefulness in poultry and swine rations, and for the recently reported implication of cottonseed meal in the incidence of trout hepatoma which has resulted in its elimination from use in fish feeds in certain areas. Processing methods must be devised for the commercial production of meals that can be fed to broilers, laying hens and swine, safely and without restriction. In order to lay the necessary groundwork for advances in cottonseed research on food, feed and industrial products and processing technology, additional fundamental information is also

needed on the chemical composition and properties of cottonseed and of various cottonseed products.

USDA PROGRAM

The Department has a continuing long-term program involving organic chemists, physical chemists, analytical chemists, biochemists, chemical engineers, physicists, and microbiologists engaged in both basic and applied studies on cottonseed and its products to develop new or extended uses for these materials.

Research to develop fundamental information on the chemical composition and properties of cottonseed products is conducted at New Orleans, Louisiana, as a basis for efficient applied research in the fields of food, feed, and industrial products from cottonseed. Some important phases of current work involve research on the problem pigments of off-color cottonseed oils; and on the chemical, physical and biochemical properties of cyclopropene fatty acids and other cottonseed constituents. The Foundation for Cotton Research and Education contributes towards research on the isolation and characterization of cyclopropene ring fatty acids of cottonseed. The National Cottonseed Products Association supports a Postdoctoral Research Associateship for conducting pioneering research on cottonseed and cottonseed constituents. Additional research on chemical composition and physical properties is carried out under contract at the University of Tennessee, Knoxville, Tennessee, on investigations of gossypol esters and mild oxidation products of gossypol and gossypol derivatives; at the University of Illinois, Urbana, Illinois, on investigation of the chemical and physical properties of cyclopropene fatty acids in cottonseed; and at Purdue Research Foundation, Lafayette, Indiana, on fundamental investigations of chemical transformations of olefinic compounds of fats and other agricultural materials by hydroboration and subsequent reactions to develop basic information for the production of useful products.

New and improved food products and processing technology are developed in research conducted at New Orleans, Louisiana. Research on confectionery fats is cooperative with the National Confectioners' Association who maintain a Fellowship at the Southern Regional Research Laboratory, New Orleans, Louisiana, in partial support of the work, and evaluate promising research products. The Office of the Surgeon General supports research to develop fat emulsions for intravenous alimentation. This research is conducted in cooperation with the Louisiana State University Medical School, New Orleans, Louisiana, and several other research groups. Informal cooperation is maintained with industry in connection with the research on new and improved food products and processing technology. Additional research on new and improved food products and processing technology is conducted under contract at the University of Illinois, Urbana, Illinois, on chemical investigations of cyclopropenoids to develop practical means of eliminating or physiologically inactivating the cyclopropenoid constituents of cottonseed oil.

Research is carried out at New Orleans, Louisiana, to develop new and

improved feed products and processing technology for cottonseed. Investigations are in progress to isolate and identify the physiologically active constituents in cottonseed meals that adversely affect the utilization of the meal as a protein supplement in nonruminant feeding. Animal tests in connection with the overall research program are conducted through the cooperation of nutritionists in State Agricultural Experiment Stations and the Animal Husbandry Research Division. The Pharmacology Laboratory at the Western Regional Research Laboratory, Albany, California, cooperates by conducting small-animal studies to determine the physiological and pharmacological effects of cyclopropene acids, gossypol and gossypol derivatives. In research directed toward providing a basis for the ultimate commercial production of cottonseed meals that can be fed to swine and poultry without restriction, as well as to ruminant animals, cooperation is maintained with the National Cottonseed Products Association, members of the cottonseed industry, and nutritionists in public and commercial agencies.

Research to develop new and improved industrial products and processing technology is conducted at New Orleans, Louisiana. Informal cooperation is maintained with industrial firms for the evaluation of promising research products for specific end uses. Additional research on new and improved industrial products is being carried out under contract at the University of Arizona, Tucson, Arizona, on the polymerization of reactive chemical intermediates derived from cottonseed oil and other agricultural materials to produce polymers having potential industrial utility.

Other research on chemical composition and physical properties is in progress under grants of P. L. 480 funds to the following foreign institutions: British Food Manufacturing Industries Research Association, Leatherhead, Surrey, England, for fundamental studies of the fatty acid and glyceride composition of cottonseed oil and the crystallizing behavior of some of the major components (project duration - 4 yrs.); University of Bombay, Bombay, India, for a study of the relationship of substituent fatty acid groups on the physical properties of diacid triglycerides of palmitic and stearic acids as a means of increasing the utilization of cottonseed oil for food and industrial purposes (project duration - 5 yrs.); Israel Institute of Technology, Haifa, Israel, for investigation of π-complexed organometallic compounds derived from polyunsaturated fatty acids to obtain fundamental information needed in expanding the utilization of cottonseed oil (project duration - 5 yrs.); and University of Rome, Rome, Italy, for basic investigations on the physical and physicochemical properties of cottonseed proteins (project duration - 5 yrs.). Additional research to develop new and improved industrial products and processing technology is in progress under grants of P. L. 480 funds to the following foreign institutions: University of Montevideo, Montevideo, Uruguay, for research on the preparation, characterization, and evaluation of derivatives of gossypol for use as biologically active materials, ultraviolet absorbers, and other products (project duration - 5 yrs.); Indian Institute of Science, Bangalore, India, for studies of the addition of carbenes to unsaturated fatty materials derived from cottonseed oil to provide possible new outlets for utilization of the oil

(project duration - 5 yrs.); and National Chemical Laboratory, Poona, India, for investigation of the synthesis and properties of new-type glycol mono alkyl ethers for control of water evaporation to extend the industrial utilization of cottonseed oil (project duration - 5 yrs.).

The Federal in-house scientific effort devoted to research in this area totals 49.9 professional man-years. Of this number 16.9 is devoted to chemical composition and physical properties, 16.1 to new and improved food products and processing technology, 14.7 to new and improved feed products and processing technology, and 2.2 to new and improved industrial products and processing technology. The contract research involves an additional 4.0 man-years, 2.4 being on chemical composition and physical properties, 0.9 on new and improved food products and processing technology, and 0.7 on new and improved industrial products and processing technology. P. L. 480 research involves 7 grants, of which 4 are on chemical composition and physical properties and 3 on new and improved industrial products and processing technology.

The following line of work was terminated during the year: (1) Investigation of the preparation and properties of alkyl aryl ketones and their derivatives from vegetable oils for industrial applications (P. L. 480 project), (under new and improved industrial products and processing technology).

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemical, Physical, and Biological Properties and Structural Factors of the Proteins. The composition, properties, structural factors and reactions of oilseed proteins and associated materials are being investigated in a program of pioneering research conducted by the Seed Protein Pioneering Research Laboratory. The fundamental information developed should lead to new concepts and possibly new applications for oilseed proteins, including cottonseed protein. Since peanuts were found to be an especially suitable experimental material and employed for much of the early pioneering research on seed proteins, the report of progress in the research is given in Area No. 7, "Peanuts Processing and Products," as in the previous report.

2. Chemical and Physical Properties of Pigments and Minor Constituents Including Cyclopropene Fatty Acids. Further progress has been made in developing information on the nature and possible origin of the problem pigments responsible for the objectionable reddish coloration in off-color cottonseed oils. Basic research on the problem pigments has led to elimination of the possibility that they arise through the route of interesterification with gossypol. Evidence was obtained that anhydrogossypol may be involved in their production. The problem pigments are lost when efforts are made to remove the glycerides by mild alkaline hydrolysis, but apparently the triglycerides may be removed from the pigments via lipolysis.

A better insight into the properties of the problem pigments was gained in the recent research, since it now appears that the pigments are not fatty esters of gossypol. A Research Associate of the National Cottonseed Products Association assisted with the research. (S4 1-96).

Contract research was initiated at the University of Tennessee on gossypol esters and mild oxidation products of gossypol and its derivatives. Nuclear magnetic resonance spectra of gossypol and several derivatives, obtained in the research have led to the following conclusions: (1) Gossypol in dioxane and in chloroform solution is apparently completely in the tautomeric form having the aldehyde group; (2) Two OH groups of gossypol are intramolecularly hydrogen bonded while the other four are not. (It is suspected that the peri OH groups are bonded to the aldehyde carbonyl); (3) The "white forms" of gossypol hexamethyl ether and gossypol hexaacetate are derived from the hemiacetal tautomeric form of gossypol; (4) Gossypol anils are in the tautomeric form having the -CH = N- grouping, rather than the other two types. The information being developed in the research will be of value in studies of the nature of the problem pigments in off-color cottonseed oil and to the problem of gossypol complexes in cottonseed meals. (S4 1-103(C)).

Several lines of fundamental investigation on the cyclopropene acid constituents of cottonseed oil have continued in in-house research. Selected organic and inorganic acids are undergoing evaluation for possible use in heat treatment of cottonseed oil to destroy the cyclopropene groups. Preliminary results are encouraging. Initial laboratory experiments to isolate methyl malvalate were unsuccessful, but work is continuing to develop methods for isolating cyclopropene acids or their derivatives. A pilot plant set up has been installed for use in preparing sizeable quantities of methyl malvalate for study. In attempts to prepare an appreciable quantity of pure methyl sterculate from Sterculia foetida oil using urea clathration and fractional crystallization from solvents, fractions containing about 98% of cyclopropene fatty acids (by gas chromatography) were obtained, but none consisted of pure methyl sterculate. Several hydrogenations were carried out with a concentrate of methyl sterculate, and the reaction products are being studied. In work on analytical procedures for cyclopropenoid fatty acids, refinement of the previously developed hydrogen bromide titration method and development of a new colorimetric method has made it possible for the first time to determine accurately the cyclopropenoid (Halphen) acid content of a number of commercial cottonseed salad oils (0.04 to 0.44%) and crude cottonseed oils (0.54 to 0.93%). Using the procedures, it has been found that in processing cottonseed oil the greatest loss of cyclopropenoid acid occurs during deodorization; and in hexane extraction of cottonseed, the last fraction of oil extracted contains a much higher percentage of cyclopropenoid acid. The Foundation for Cotton Research and Education contributes towards the research on cyclopropene fatty acids of cottonseed. (S4 1-105).

Initial phases of contract research at the University of Illinois on the chemical and physical properties of cyclopropene fatty acids in cottonseed

have resulted in the isolation of methyl sterculate by transesterification of the glycerides from Sterculia foetida. The ester was characterized by its nuclear magnetic resonance spectrum. Infrared spectral data have been compared with Halphen data as a means of quantitative assay of sterculic acid, and a study to determine the products of the Halphen reaction was initiated. Cyclopropylamine and dimethylpyrazoline have been prepared as intermediates to model compounds in the synthesis of sterculic acid, a homologue of malvalic acid. The present approaches will be continued to obtain basic information on the properties of the cyclopropene acids important to improving cottonseed oil and meal and to developing methods of analysis of these acids in oil and meal. (S4 1-104(C)).

3. Chemical, Physical, and Physiological Properties of the Oil and Fatty Acids. Chemical work on the quality evaluation of alumina bleached cottonseed oils was completed. Neither activated alumina nor sulfurous acid-treated alumina induced significant amounts of polymerization or isomerization as compared with conventional earth-bleached oils. Hydrogenation proceeded normally, and organoleptic stability was satisfactory. Preliminary indications are that, although bleaching of cottonseed oil with sulfurous acid-treated alumina eliminates the response to the Halphen test, the oils are physiologically active, as determined from feeding tests with laying hens; and the Halphen test, which has been assumed to be specific for cyclopropene fatty acids, is apparently not a reliable test for the presence of physiologically active substances in cottonseed oil. Eggs produced by hens fed the various oils are being examined for effects on pH of yolk and white, gossypol-formed pigments in the yolk, and fatty acid distribution in yolk fats. (S4 1-96).

Feeding tests are being conducted in an effort to determine if malvalic acid in refined and bleached cottonseed oil of commerce constitutes a problem for the cottonseed industry. Comparison of the properties of eggs and egg fats from hens fed cottonseed oils treated to eliminate response to the Halphen test with those from hens receiving strongly Halphen-positive cottonseed oil showed that the stearic:oleic acid ratio was higher in the cottonseed oil-produced eggs than in corn oil-produced eggs; but the high ratio does not seem to be dependent on the Halphen-positive constituent in cottonseed oil. Studies with rats fed Halphen-positive and Halphen-negative cottonseed oils reveal that heart and brain fatty acid distribution are identical for male and female rats and for all oils studied. The difference between cottonseed and corn oil shows up in the liver fat, and the difference is probably not due to the Halphen-positive constituent of cottonseed oil. Louisiana State University and the Pharmacology Laboratory of the Western Division cooperate in conducting the animal studies. The Foundation for Cotton Research and Education contributes towards the research. (S4 1-105).

In P. L. 480 research at the British Food Manufacturing Industries Research Association, studies are in progress on the fatty acid and glyceride composition of cottonseed oil and the crystallizing behavior of some of the major

components. The fatty acid composition of a number of oils from cottonseed of various origins and processing histories have been examined by four different methods. Gas-liquid chromatography yielded results nearest to the accepted true values. Fractionation by low temperature crystallization has indicated that, although cottonseed oil contains 3 major component fatty acids, only 4 out of 26 probable triglycerides occur to the extent of over 8%, and the minor component acids are very uniformly distributed throughout the glyceride components of the oil. Further progress in the research is expected to provide data useful in the selection and processing of cottonseed oils for the commercial production of improved salad oils in optimum yields. (UR-E29-(40)-26).

In P. L. 480 research at the University of Bombay, studies are being made of the relationship of the substituent fatty acid groups to the physical properties of diacid triglycerides of certain saturated fatty acids, including those that occur normally in cottonseed oil. The diacid triglycerides that are of interest in this work are those containing one or two molecules of palmitic or stearic acid, and two or one of even-carbon saturated fatty acids of the series from acetic to stearic acid. Progress is being made in determining the properties of these synthesized diacid triglycerides and their binary mixtures. The information obtained is expected to provide the basis for the further development of fats and oils specifically tailored for special food and industrial end uses. (UR-A7-(40)-3).

B. New and Improved Food Products and Processing Technology

1. New Edible Oil Products Including Confectionery Fats, Food Coatings and Other Specialty Products. Several esterification processes have been investigated for producing cocoa butter-like fats. Direct esterification of diglycerides of palmitic and stearic acids with oleic acid, in a manner to minimize interesterification, gave up to 90% yields of refined cocoa butter-like fat in small scale laboratory experiments. Use of stannous chloride as catalyst produced lower yields but less color (water-white products) than with p-toluenesulfonic acid. When the direct esterification was scaled up considerably, yields were not as good primarily because of difficulty in satisfactory removal of water formed in the reaction. Best results were obtained when the disaturated diglyceride was esterified with oleic acid under rapid agitation and a partial vacuum. A yield of 68.2% oleodisaturated glycerides was obtained, based on the unfractionated, refined fat.

Yields of 50 to 70% oleodistearin were obtained in the refined products produced by an esterification process involving addition of mono-olein in solvent to stearic acid under conditions in which the water produced is removed by boiling solvent.

In a third esterification process, oleic acid was reacted with acetic anhydride and these mixed anhydrides were used to esterify diglycerides of palmitic and stearic acids. The best products contained about two-thirds oleodisaturated triglycerides, with some of the remainder monoacetoglycerides.

A quantitative method for determining percentage of trisaturated, monounsaturated disaturated, diunsaturated monosaturated, and triunsaturated glycerides in esterification products has been developed. It should be very useful in the confectionery fat research.

The rapid tempering of confectionery fats and chocolate by intense mechanical working was investigated further using mostly liquid products. Both cocoa butter and chocolate were found to convert to the highest melting form when the rapidly cooled, unseeded liquid product was worked mechanically. This conversion was most rapid when the liquid products were only a few degrees above the melting points of the α-polymorphic form. The rapid tempering process has received considerable industrial interest and is being evaluated on a pilot-plant scale by an industrial concern. The tempering time of chocolate and similar fats can be reduced from several hours to a few minutes with the new process. The research on confectionery fats is supported in part through a Fellowship sponsored by the National Confectioners' Association. (S4 1-91).

Research has continued on development of new polyester products from cottonseed oil. Several types of reactions have been compared for feasibility in preparing amylose esters of long-chain fatty acids, products which have attracted considerable industrial interest. Selection of a suitable solvent system to dissolve amylose as well as the ester has presented some difficulty. The most promising reactions investigated appear to be esterification in dimethylsulfoxide and interesterification in acetonitrile. The preparation of amylose palmitate and amylose oleate using acid chlorides is in progress. These esters will be tested as plasticizers for amylose films by a commercial firm.

Methods have been discovered for preparation by direct esterification (using p-toluenesulfonic acid catalyst and continuous removal of water of esterification by azeotropic distillation) of good yields of diglycerides and monoglycerides. For example, simple esterification of 1-monostearin with oleic acid at 80°C. yielded as much as 72.3% of diglycerides, and esterification of glycerol with stearic acid at 100°C. yielded up to 70.1% monoglycerides, each calculated on a glyceride basis. The discovery of this process will form the basis for the development of new fat products using monoglycerides or diglycerides as intermediates.

In further investigations of fungistatic activity of fats and fatty acids derived from cottonseed oil, the compounds, tricaproin, oleodicaproin, methyl caproate and caproic acid showed some fungistatic activity against the micro-organism, P.roqueforti, under the liquid medium test conditions employed. Triglycerides of heptanoic, octanoic, nonanoic, decanoic, undecanoic, undecenoic, and dodecanoic acids have been prepared for testing of their fungistatic activity. (S4 1-90).

Improved fat emulsions for intravenous alimentation have been developed in research supported by the Office of the Surgeon General and conducted

cooperatively with the Louisiana State University Medical School and several research groups. Extensive evaluations on test animals of the SR fat emulsion 695, which contains an improved primary emulsifying agent, gave excellent results. Limited clinical tests of this SR emulsion prepared by a pharmaceutical company show promise, but the commercially prepared emulsion did not have as good storage stability as that prepared at the Southern Division. The emulsion was fortified with vitamins, minerals, and protein hydrolysates, which has not been feasible with conventional emulsions because of their physical instability upon addition of the hydrolysates, and administered to animals with good results. Preliminary to the next large-scale preparation of emulsion 695 for testing in humans, each component of the emulsion has been prepared in emulsified form and distributed to the Sub-Task Group for testing in rabbits.

Other promising emulsions have been prepared on a laboratory scale. In some of these the two standard emulsifiers are used at 50% of their usual concentration and the patented emulsifier omitted; in others, new type emulsifiers replace the patented emulsifier component at the same level in the standard emulsion (SR 695). Cottonseed and soybean oil emulsions stabilized with phosphatidyl choline emulsifier have also been prepared for testing in rabbits.

A synthetic triglyceride made from molecularly distilled methyl esters of cottonseed oil fatty acids was used in preparation of an emulsion for studies of the cause and nature of liver pigmentation resulting from prolonged administration of cottonseed oil emulsions. (SG-0-1).

2. Processing Technology Related to Improved Oil Products, Including Modifying or Eliminating Cyclopropene Acids in Cottonseed Oil. Recent engineering studies to develop a commercial process for preparing cocoa butter-like fat from cottonseed oil have been concerned primarily with determining the effects of short crystallization time, an important consideration in a continuous process for producing cocoa butter-like fat. Experiments with a vertical batch laboratory votator indicated that short time crystallizations without crystal modifiers can be used to separate the objectionable saturated fats in the fractionation of interesterified fats in solvent in the preparation of cocoa butter-like fats. In investigations of subsequent steps in the process, bench-scale experiments were conducted to determine the effects of various crystal modifiers on decreasing crystallization time of the crude cocoa butter-like fat product from fat-acetone solutions, and on improving the purification step. Saturated fats separated during the first crystallization offer attractive economic possibilities for use as a modifier. Using this modifier, crystallization times were decreased from about 40 minutes to less than 5 minutes, and yields of crude cocoa butter-like fat were about 80% of those obtained by the original slow crystallization procedure. In the purification step, it was found that either commercial pentane or hexane can be employed as solvent, with a commercial crystal modifier, to give a shortened crystallization time (60-100 seconds range) sufficient for satisfactory separation of

high melting fat impurities. The present lines of work will continue. (S4 1-101).

In further development work on the new process for bleaching off-color cottonseed oils with activated alumina, optimum operating conditions for the process have been established. The filtering problem has been substantially solved by the use of filter aid (either cellulose fiber or pearlite) with an alumina of a specific particle size composition. Improving the dispersion of the alumina in the oil by the use of intensive mixing to thoroughly break up agglomerated alumina particles gave markedly lighter oil colors, indicating a possible reduction in the quantity of alumina required. Oil losses by alumina bleaching, when the cake is washed with hexane while still in place on the filter, were found to amount to 0.8% or less of the input refined oil. This oil loss is significantly lower than the loss by re-refining followed by earth bleaching, as in conventional processing.

A pilot-plant unit capable of bleaching 200-pound batches of cottonseed oil with activated alumina has been installed. It will be operated to obtain material balance data needed to finalize costs of the alumina bleaching process, and to produce spent alumina for regeneration studies. A method for pre-determination of the dosage of alumina required to bleach a given refined oil to a desired color has been found. All three of the major alumina producers now believe they can commercially produce the required alumina at 8 to 10 cents per pound, which would remove the major obstacle to commercial use of the new bleaching process. A major producer and processor of cottonseed oil is considering use of the alumina process commercially and has initiated engineering studies towards this end. (S4 1-92).

Experiments have shown that hydrogenation can preferentially destroy cyclopropene acid groups in cottonseed oil without forming significant amounts of positional and geometrical isomers of other unsaturated fatty acid groups. Optimum conditions were 105-115°C., 20 p.s.i.g., 0.1% nickel, and a poor rate of hydrogen dispersion. With an iodine value reduction of only 1.7 units, the 0.4% malvalic acid groups were hydrogenated completely, and only 2.1% of <u>trans</u> isomers formed. The oil was more easily winterized with the same yield. This could be a very practical method of improving cottonseed oil quality if it should be established that cyclopropene acids are undesirable in an edible oil. Feeding tests will be necessary to establish whether the hydrogenation has eliminated the physiologically active cyclopropene acids. (S4 1-105).

Contract research is being initiated at the University of Illinois for chemical investigations of cyclopropenoids to develop practical means of eliminating or physiologically inactivating these type constituents present in cottonseed oil without affecting the oil's edible properties. (S4 1-107(C)).

C. New and Improved Feed Products and Processing Technology

1. Basic Research to Improve Nutritive Value of Cottonseed Meal for Poultry and Swine, Including Investigations of Physiologically Active Constituents. Studies were continued on the relationship between cottonseed protein amino acids and the nutritive quality of cottonseed meals. Nutritional data for commercial cottonseed meals indicate that the influence of lysine, which is limiting in cottonseed meal-corn rations, is so great that the contributions of other constituents to the total variance in growth and feed efficiency is completely masked. Therefore, the nutritive quality of presently available commercial cottonseed meals can be assessed through a determination of available lysine.

Improvements have been made in the previously developed dinitrophenylation method for quantitative estimation of available lysine in cottonseed meals and other vegetable proteins. It was demonstrated that the time for acid hydrolysis can be reduced to 6 hours, the ratio of aqueous HCl to meal should be at least 400:1 to minimize errors due to destruction of lysine, and the level of carbohydrate may be as high as 20% without causing error in the assay. Lysine has been found to be the only amino acid of cottonseed proteins surviving HCl hydrolysis that is affected by commercial cottonseed processing conditions. The amino acid composition of cottonseed meals, before and after dinitrophenylation, has been compared. With the exception of lysine, the amino acid composition of the untreated meals was found to be relatively constant, within limits of measurement, over a wide range of meals produced in different areas from seeds of diverse varieties. Tryptophane and cystine-cysteine have not been investigated. In the dinitrophenylated meals, there were reductions in serine, methionine and lysine and almost complete loss of histidine and tyrosine.

Research studies of the relationship between amino acids of cottonseed protein, especially tryptophane and methionine, will be continued. (S4 1-95).

The characteristics and physiological properties of various fractions of cottonseed pigment glands were investigated as a possible basis for further improvement of the nutritional quality of cottonseed meals. A water-dispersible fraction (accounting for 81% of the glandular weight and containing 21% gossypol) and a water-insoluble fraction (containing 8% gossypol) have been separated from the glands. The amino acid pattern of the protein present in these two fractions is different from that of parenchymatous seed tissue. The protein efficiency of glandless cottonseed meal and the availability of lysine are both decreased when the meal is mixed with the water-dispersible gland fraction. It has become evident that the large reduction in protein efficiency observed cannot be attributed solely to gossypol per se which accounts for only 21% of the water-dispersible gland fraction.

In another phase of work, studies with Halphen-positive and Halphen-negative

cottonseed oils indicated that both cause an increase in the stearic:oleic acid ratio in the depot fat and liver fat of rats receiving these oils (15%) as compared with the effects of corn oil. It is not known if the effect is physiologically significant. The fatty acid pattern of the depot fat was very similar to that of the ingested fat, while the liver fat contained much higher levels of stearic acid. (S4 1-97).

Research has been initiated to isolate and identify the physiologically active constituents in cottonseed meals that cause mortalities among swine, to develop information for producing cottonseed meals that can be used without restriction in feeding of nonruminants. Chemical analyses of tissues from rats fed acetone-hexane-water extracted meals at the Western Division are being conducted at the Southern Division. Feeding tests with cottonseed meal known to kill pigs are being initiated at the Western Division. Efforts will be made to isolate the factor responsible for abnormalities that may be noted in the tissues. (S4 1-110).

2. Processing Technology Related to Improved Meals. Pilot-plant investigations of cottonseed processing using hexane-acetone-water solvent mixtures were continued to develop a process suitable for use on a commercial scale. Results to date with a continuous, 8-cell, pilot-plant basket extractor, simulating commercial extractors, indicate that cottonseed meals superior to those currently produced commercially, and oil of high quality, can be produced by processing with hexane-acetone-water mixed solvent using conventional basket type extractors. Investigation of selected mixed solvent compositions showed that a fairly wide range of hexane-acetone-water compositions (39/57/4, 60/39/1, 25/74/1 and 25/73/2) gave satisfactory results.

In recent work a continuous, total immersion, inclined, countercurrent extraction device was designed and put into operation as an auxiliary to the basket extractor to reduce the excessively long extraction time required. Best overall results were obtained when used as a post extractor with a solvent composition of hexane-acetone-water of 60/39/1. Using this combination of extractors, satisfactory extraction was obtained when employing a cycle of only 40 minutes in the basket unit and 40 minutes in the auxiliary unit. This promising approach will be investigated further using a horizontal immersion device. The present integrated pilot plant will be operated to produce at least one ton of acceptable meal for extended feeding tests and provide engineering data for material balances and cost studies.

A second pilot-plant approach for the mixed solvent extraction of cottonseed--10-stage countercurrent extraction with an 18" diameter vibrating screen separator--has been continued. Experiments have indicated that it would be possible to process about 12 tons of cottonseed per 24 hour day with the unit, with a residence time in each separator of about 17 seconds. The fines problem is disposed of by recirculating the fines from the final miscella to step 5 in the 10-step countercurrent extraction procedure. Feeding tests on meals produced by this process showed that they had

protein efficiencies comparable to soybean meal.

Limited exploratory bench-scale tests employing mixed solvent with a modified filtration-extraction process, wherein cottonseed linters or hull fibers are used as a filter aid, gave promising results. Use of this technique could lead to a broadened application of the process to many other oleaginous materials otherwise difficult to extract. (S4 1-94; S4 1-95; S4 1-111).

D. New and Improved Industrial Products and Processing Technology

1. Basic Research to Develop New Reactions and Products Suitable for Industrial Use. Approximately 60 additional fatty amide derivatives, mostly N-acyl derivatives of 5-, 6- and 7-membered cyclic imines, have been synthesized, characterized, evaluated as polyvinyl chloride plasticizers, and submitted for antimycotic activity screening. Most of these were piperidides and alkylpiperidides. The piperidides and alkylpiperidides of long-chain fatty acids have been found to be superior in compatibility as plasticizers for polyvinylchloride resins to any of the amides examined to date. They show particular promise costwise since they can be made from cheap or potentially cheap raw materials. In general they are highly efficient, primary plasticizers exhibiting good low temperature characteristics. The N-oleoylpiperidine mixture prepared from a low-priced commercial byproduct mixture of alkylpiperidines imparted very good plasticizer properties, and also proved to be an acceptable softener for nitrile rubber. Soil burial tests (155 days) on specimens of Vinylite VYDR plasticized with N-oleoyl derivatives showed that the antimicrobial activity increased in the order morpholide, piperidide, 2-methylpiperidide, mixed piperidide (from a mixture similar to the byproduct mixtures of alkylpiperidines), 2,6-dimethylpiperidine, DOP. Only the last two showed no discoloration (pink stain) at the end of the test period.

Considerable industrial interest has been shown in these piperidide plasticizers by manufacturers of plasticizers and fatty acids and by producers of potentially cheap piperidine and alkylpiperidine mixtures. One commercial firm is preparing pilot-plant batches of fatty acid piperidides for market evaluation. (S4 1-99).

Research under a P. L. 480 grant, recently expired, has been completed at the Institute for Research on Oils and Fats. These investigations were concerned with the preparation and properties of alkyl aryl ketones and their derivatives from vegetable oils for industrial applications. The research has produced a number of chemical derivatives of the alkyl aryl ketone type that have potential utility in such industrial products as fungicides, lubricants, plasticizers, and surface-active agents. (UR-E9-(00)-29).

Good progress is being made in P. L. 480 research at the University of Montevideo in the preparation, characterization and screening of a number

of derivatives of gossypol having potential industrial utility. Several new imino derivatives (anils) have been prepared. Reduction of these to substituted amines is expected to yield derivatives having ultraviolet screening or antioxidant properties. Derivatives that have been prepared by reaction with hydantoin would be expected to exhibit physiological activity or catalytic activity in certain organic reactions. Screening for such potential uses is being considered. (UR-S9-(40)-2).

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2/ Publication resulting from research under grant of P. L. 480 funds to the foreign institution.

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AREA NO. 7 - PEANUTS PROCESSING AND PRODUCTS

Problem. Peanuts constitute a major cash crop in the Southern States and are in surplus. Because of the high price of peanuts in the United States, peanuts are used almost exclusively (approximately 73 percent of the crop) in foods such as peanut butter, confections, and roasted and salted nuts. New type food products and improvement in the quality and uniformity of existing products are needed to increase consumer acceptance and extend markets; the average per capita consumption has been rather stable since World War II. The increased trend toward mechanical harvesting has necessitated the use of artificial means for curing and drying peanuts, with the result that processed peanuts and peanut products do not always possess the same desirable flavor and physical properties as peanuts which have been cured slowly in the field. Information is needed as to the physical and chemical characteristics of those chemical constituents in peanuts which affect the properties of processed products as a basis for developing new or improved products and processing procedures. Fundamental studies of peanut protein and associated materials could similarly lead to the development of new concepts and new uses.

USDA PROGRAM

The Department has a continuing long-term program involving organic chemists and biochemists, engaged in basic studies on peanuts and peanut products to increase consumer acceptance and extend markets for peanuts.

Research to develop basic information on the chemical composition and properties of peanuts, its constituents, and processed peanut products is carried out at New Orleans, Louisiana. As a part of the Seed Protein Pioneering Research Laboratory's research on various seed proteins, fundamental investigations of peanut proteins and associated materials are conducted to form the basis for developing new concepts and perhaps new uses for peanuts and peanut proteins. In other in-house research, peanut constituents and their modification by processing that influence nutritive properties and consumer acceptance of processed peanut products are studied. A recently initiated phase of this research involves investigations of the proteins and nonglyceride lipid-soluble constituents of peanuts and processed peanut products. The Crops Research Division of ARS and several State Experiment Stations, including Georgia, Alabama, and Texas, cooperate in the research by providing samples of peanuts of known variety and of known growing, harvesting, and drying histories. Louisiana State University cooperates by conducting evaluation tests on selected peanut isolates. Additional research on chemical composition and properties is being carried out under contract at Evans Research and Development Corporation, New York, N. Y., on the isolation, identification and characterization of flavor and aroma components of processed peanut products to form the basis for producing improved peanut products of greater consumer acceptability.

The Federal in-house scientific research effort in this area totals 5.9 professional man-years. All of the present effort is on chemical composition and physical properties. The contract research involves an additional 1.0 man-years, all of the effort being on chemical composition and physical properties.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemical, Physical, and Biological Properties and Structural Factors of the Proteins. The composition, properties, structural factors and reactions of the proteins and associated materials of various seeds, including peanuts, are being investigated in a program of pioneering research conducted by the Seed Protein Pioneering Research Laboratory. Recent research has made significant contributions toward (1) a new classification of seed proteins, (2) new tools for the study of proteins, (3) a better understanding of the role of protein particles in seeds, and (4) elucidation of the role of lipolysis in mobilization of lipids in germinating seeds.

A major objective in the work has been to examine existing methods of classifying seed proteins and to provide the basis for development of new classifications which would have more meaning in terms of the function of the proteins in seeds. The present classification of seed proteins has been based upon solubility. This is artificial since it does not relate to any biological function of the proteins and has become increasingly unreliable as newer methods of analysis of proteins in mixtures, such as column chromatography and gel electrophoresis, have shown the various protein fractions to be grossly impure.

As the result of research in the Seed Protein Laboratory and other laboratories there now exists the basis for a new classification of seed proteins. It has been shown clearly that proteins exist in subcellular particles in seeds. In some seeds such as peanuts, soybean, and peas these particulate proteins comprise the majority of the protein. In others, such as wheat, they probably comprise a major portion of the proteins but not the majority. A uniqueness of seeds is that they contain a substantial amount of these particle-bound proteins. The new classification, based on protein bodies, provides a basis for simpler sources of the proteins and for eliminating the possibility of formation of artifacts during grinding of the entire tissue; for concentrating some of the components which are minor in the entire seed but are major components of subcellular particles; and it brings the field of seed proteins into the general field of seed biochemistry.

The Seed Protein Laboratory research has demonstrated clearly, both on the light and electron microscope level, the presence and morphology of protein bodies in peanuts and cottonseed. Moreover, by at least three different techniques, protein bodies were isolated from these two seeds and shown to

contain proteins, and even in some instances to contain fractions of the total proteins. The bodies not only stained for protein but were shown by isolation and chemical analysis to contain proteins.

As the point about subcellular distribution of seed proteins is now clearly established, research emphasis is being placed on (1) the isolation of pure seed proteins from particles, and (2) the biochemistry of the protein particles, and their role in development and germination of the seed.

New tools for the study of proteins have been developed in recent work. Included are new equipment and techniques for column electrophoresis on polyacrylamide gel, and for calorimetry.

In the previous report it was pointed out that polyacrylamide gel (Cyanogum gel) is very discriminating in separating protein components which are highly interacting. Electrophoresis on this gel of α-conarachin, shown pure by chromatography on DEAE cellulose and by sedimentation, indicated that it still contains about 10 to 15% impurities. It, therefore, was deemed advisable to develop equipment for more quantitative analytical analysis by electrophoresis on polyacrylamide gel and, eventually, for preparative electrophoresis using this medium. Equipment has been designed and built for analytical electrophoresis on a column of polyacrylamide. The protein is continuously eluted off the bottom of the column and is monitored by ultraviolet absorption. Bovine serum albumin was the model for development of this apparatus. This technique clearly shows the separation between the monomers and the dimers of bovine serum albumin; there is a strong possibility that it will also separate several of the monomers of this compound, which have defied separation heretofore. A design has been made of a multicolumn apparatus which will make possible preparative as well as analytical electrophoresis. It is the first time that it has been possible to observe electrophoresis in a medium through which no liquid will flow. It makes possible the study of the interesting properties of a completely crosslinked gel.

A microcalorimeter capable of detecting a change of one micro degree centigrade equivalent to one millicalorie has been constructed and is operating. It is intended for study of the structures of seed proteins. But first it is necessary to study the performance of this instrument on a well studied biological model. In cooperation with Dr. Eraldo Antonini and Dr. Jeffrey Wyman of the University of Rome, experiments are being conducted on the heat of protonation of human hemoglobin at various pH values. The results of this work have already attracted wide interest and are being reported at an international conference on calorimetry in Lund, Sweden.

Research has been conducted to develop information on the role of the protein particles, that is, to relate them to other biochemical properties of the seed. One major question involving oilseeds is the location of the lipids. It has generally been held that these are spread throughout the

cytoplasm of the parenchyma cells. Careful electron microscopy following staining designed to maintain lipids in place has now disclosed that lipids of the cottonseed are in two locations: within protein particles and in interparticle locations. These conclusions were reached by observing dense osmiophilic regions within the protein bodies and in the interbody space. They were confirmed by isolation of two particles from cottonseed cotyledons - a dense and light particle, both containing lipid. The isolation was accomplished by an entirely new approach of fixing the particles with tannic acid and then subjecting them to density separation. The heavy particles contain up to 30% lipids and the light ones up to 60%. It is now clear that the protein bodies are not completely protein-containing but are also associated with other material such as lipids. In previous reports it was pointed out that phytic acid was also located in some protein particles.

Another question was whether the resting seeds contain mitochondria. Mitochondria had been found in germinating seeds and in seeds which had been moistened for 12 hours, but had not been reported in the dry resting seeds. Suitable techniques of staining have now clearly shown that mitochondria are present in the resting seeds; some clusters of mitochondria under other staining techniques might even have been mistaken for protein bodies. There are, therefore, clusters of materials containing proteins which are not clearly protein bodies and which contain mitochondria and presumably other biologically-active membranous structures.

The association of proteins with storage materials and membranous structures might suggest that many of the proteins may have had an enzymatic function in the synthesis of storage material. The proper selection of enzyme tests might even show in the mature seed the vestiges of these enzyme systems. A second possible function for the proteins might be to serve as a matrix for the deposition of enzymes and other materials, similar to the function of structural protein of ribosomes, chloroplasts, or mitochondria. Another possibility is that some, at least, of the protein bodies represent organized systems of enzymes and substrates which participate in the synthesis of new enzymes in the early stages of germination. And finally there still may be some proteins which truly are reserves. Most proteins disappear from the storage tissue on germination. No matter what their function may have been initially, the same protein also serves eventually as a source of amino acids and nitrogen for the growing tissue of the seedling.

Further investigations have been made on the role of lypolysis in mobilization of lipids in germinating seeds. It is generally considered that lypolytic activity starts early in germination and that relatively large quantities of free fatty acids are formed. This information is based on rather flimsy evidence which includes titration of total acidity in germinating seeds without efforts to separate out the long chain fatty acids. Moreover, no efforts are made to inhibit enzymatic activity when the seed is ground to extract the free fatty acids. Therefore, it is entirely possible that the high free fatty acid content reported is actually an artifact of the method of measurement or of the method of disintegration

and extraction of the fatty acids. This is an important consideration because it has a bearing on the integration of activities in the mobilization of fats. It determines whether the lipase activity is an independent function in the seed or is closely integrated with the other activities involved in the breakdown of the fatty acids.

It has been found in the castor bean that the free fatty acid pool during germination is very low, 0.2 to 0.4 micromoles out of a possible 300 microequivalents present originally per kernel. This would mean that very little free fatty acid is present in the germinating seed at any given time. Although there is no lypolysis in the intact seed, once the seed is macerated there is very active lypolysis. This means that within the seed the system is so organized that no free fatty acids accumulate. Active work is now proceeding to determine whether the lipase may be isolated in a separate subcellular unit away from the other enzymes involved in fat mobilization.

2. Identification of Constituents and Factors Influencing Flavor, Aroma, Color, Structure, and Nutritive Value of Processed Products. Investigations have continued on the constituents of peanuts and their modification by processing that influence nutritive properties and consumer acceptance of peanut products. The myotonic factor in peanuts has been concentrated about 50,000-fold, but the pure substance has not been isolated. This material is physiologically active in concentrations of less than one part per million. Its activity is associated with a spectral absorption band at 5.65 microns. Loss of activity on mild hydrolysis is accompanied by the loss of this band, the appearance of a band at 7.4 microns, and the enhancement of the absorption band at 6.25 microns. This behavior might be accounted for by the opening of an unsaturated lactone ring to yield a compound having both acid and carbonyl groups.

A strong muscle relaxant has been isolated from the concentrates, along with additional crystalline substances that remain to be identified. Nicotinic acid and nicotinamide have been isolated and identified among the crystalline materials from the fractionation of the alcohol extract of defatted peanuts.

The use of peanut flour and peanut butter in hemophilia therapy appears to be expanding. Confirmation of the effectiveness of these peanut products for this purpose continue to be received.

The physiological tests on various peanut fractions isolated in the course of the research are carried out in the Department of Zoology at Louisiana State University. (S4 1-100).

A new line of basic research on peanuts, which will complement the work on characterization of alcohol-soluble constituents, was recently initiated. The influences of processing on the amino acid patterns of peanut proteins, as related to the properties of peanuts and peanut products, will be

determined. Non-glyceride lipid-soluble constituents of peanuts and processed peanut products will be isolated and identified. (S4 1-109).

Contract research is also in progress at Evans Research and Development Corporation, on the isolation, identification and characterization of flavor and aroma components of processed peanut products to form the basis for producing improved peanut products of greater consumer acceptability. (S4 1-106(C)).

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

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AREA NO. 8 - TUNG PROCESSING AND PRODUCTS

Problem. Tung oil has lost much of its traditional market in protective coatings to synthetic raw materials. New and improved industrial products from tung oil must be developed to recapture lost markets, maintain present markets, and provide new outlets for surplus tung oil. Basic information is needed on the chemical composition and properties of tung oil and its fatty acids, and on the chemical modification of these materials to permit more effective exploitation of their unique characteristics in protective coatings, agricultural and industrial chemicals, surfactants, and plasticizers. For example, improved coatings utilizing tung oil are needed to meet increased performance demands and competition from synthetic polymeric coatings. Intumescing fire-retardant coatings and water-reducible coatings containing tung oil are desired. A limited market of low economic value exists for tung meal as a fertilizer. Research is needed to develop more information on profitable uses of tung meal to benefit the overall economy of the tung industry.

USDA PROGRAM

The Department has a continuing long-term program involving organic chemists engaged in both basic and applied research on tung and its products. Emphasis in the present program is on development of new and improved industrial products from tung oil and its derivatives.

Research is conducted at New Orleans, Louisiana, to develop fundamental information on the chemical composition, properties, structural factors, and reactions of oilseed proteins, as a basis for development of new concepts and possibly new uses for oilseed proteins.

Research to develop new and improved industrial products from tung oil is carried out at New Orleans, Louisiana, with cooperation and support by the Pan American Tung Research and Development League and the U. S. Army Engineers Research and Development Laboratories. The League maintains a part-time Fellow for research on the production of improved protective coatings. Major emphasis is placed on the development of exterior, intumescing fire-retardant surface coatings using tung oil alkyds. The tung alkyds are being chemically altered and formulations modified to produce coatings which will intumesce to give a thick cellular, fire-resistant material upon thermal or flame exposure. The U. S. Army Engineers evaluate the more promising fire-retardant coating formulations developed with their support. Other investigations involve studies of the chemical modification of tung oil and its fatty acids to produce chemical intermediates having utility in protective coatings, and as agricultural chemicals, surfactants or plasticizers. Informal cooperation is maintained with industrial firms and other agencies for the evaluation of promising chemical intermediates for specific end uses.

Other research in the area of chemical composition and physical properties is in progress under a grant of P. L. 480 funds to the National Chemical Laboratory, Poona, India, for investigations of the effect of heat on tung oil and its derivatives, and characterization and identification of compounds resulting from heat treatments, to extend the utilization of tung oil (project duration - 5 yrs.).

The Federal in-house scientific research effort in this area totals 6.4 professional man-years. Of this total 0.9 is devoted to chemical composition and physical properties, and 5.5 to new and improved industrial products. P. L. 480 research involves 1 grant for research on chemical composition and physical properties.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Structural Factors, Properties and Reactions of the Protein. The composition, properties, structural factors and reactions of oilseed proteins and associated materials are being investigated in a program of pioneering research conducted by the Seed Protein Pioneering Research Laboratory. The fundamental information developed should lead to new concepts and possibly new applications for oilseed proteins, including tung protein. Since peanuts were found to be an especially suitable experimental material and employed for much of the early pioneering research on seed proteins, the report of progress in the research is given in Area No. 7, "Peanuts Processing and Products," as in the previous report.

B. New and Improved Industrial Products

1. Intumescing Fire-Retardant Surface Coatings from Tung Oil Alkyds. Exterior and interior, intumescing fire-retardant coatings showing considerable promise are being developed from chemically modified tung oil-containing vehicles in research supported in part by the U. S. Army Engineers Research and Development Laboratories and the U. S. Air Force, and assisted by a part-time Fellow of the Pan American Tung Research and Development League. Paint films prepared from these new-type coatings produce a thick carbonaceous mass when subjected to flame and heat, thus insulating the coated material from being engulfed with flames. This has important implications, since more than 11,000 lives are lost annually in the United States by fire and our property losses by fire amount to over one billion dollars a year. Effective fire-retardant coatings should find use in civilian as well as defense applications, and could greatly reduce this loss of life and property.

Two of the better experimental exterior, intumescing fire-retardant paint formulations (which were developed from chemically modified domestic vegetable oils, chemically modified intermediates, and commercial paint ingredients) were modified to improve their can stability, brushability, drying

characteristics, color and tint retention, water resistance, weather resistance, spumific (foam-forming) and carbonific (carbon-forming) properties. Both of these formulations exhibited good fire retardancy when screened with the mild USAERDL fire-test cabinet and with the simple but severe SU screening test. Upon evaluation in the Forest Products Laboratory 8-foot tunnel furnace, performance of the formulations was not as good as desired but quite satisfactory for the present stage of development.

Recently a new formulation ("H") was prepared, in which a greater concentration of synthesized carbonific materials was successfully incorporated. It has exhibited improved fire retardancy, such as reduced flame-spread and heat-contributed index values, in comparison with earlier formulations. Paint films of formulation H gave excellent fire retardancy when evaluated in the USAERDL fire-test cabinet, good fire retardancy in the severe SU screening test and the Forest Products Laboratory 8-foot tunnel test, but unsatisfactory performance in the Underwriters' Laboratories 25-foot tunnel test. Failure in the latter test was apparently largely due to the evolution of too much gas from the films when they were subjected to the severe flame and heat. An elementary 8-foot tunnel furnace devised at SU is now being employed to screen new experimental coatings more efficiently prior to testing them in the 25-foot furnace. Attempts will be made to improve formulation H by reducing thermoplastic flow and gas evolution, and also to improve brushability. (S4 1-98).

2. Chemical Modification of Tung Oil to Produce New and Improved Products Such as Protective Coatings, Agricultural Chemicals, Surfactants and Plasticizers. In research on chemical modification of tung oil and its fatty acids to produce materials having industrial utility, attempts have been made to improve the surface active properties of tung monoglycerides for applications as fugitive emulsifiers. Difficulty was encountered in the sulfation of eleostearate monoglycerides utilizing the pyridine-SO_3, dioxane-SO_3 and sodium chloride-SO_3 complexes as well as chlorosulfonic and fuming sulfuric acids in various solvents. Maximum sulfation was only 10% of the theoretical, and a number of side reactions occurred to give a mixture of products having reduced unsaturation and properties not desirable for the use as fugitive emulsifiers. Other approaches than sulfation will be investigated. (S4 1-93).

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

New and Improved Industrial Products

Goldblatt, Leo A. and Hopper, Lucien L., Jr. February 26, 1963. Epoxy resin esters containing tung oil fatty acids. U. S. Patent No. 3,079,354.

Rayner, Eric T. and Hopper, L. L., Jr. 1962. Some problems in formulating fire-retardant paints. Proc. 29th Ann. Conv. Am. Tung Oil Assoc. 29, pp. 18-21, 25. 1/

Root, Frank B. (Naugatuck Chemical). June 4, 1963. Compatible tung oil-unsaturated alkyd resin compositions and methods for producing same. U. S. Patent No. 3,092,596.

General

Kopacz, B. M. and Hoffpauir, C. L. 1963. Foreign utilization research on tung oil. Am. Tung News 14(1), pp. 8-9.

1/ Publication resulting from research supported by funds transferred from the U. S. Army Engineers Research and Development Laboratories and the U. S. Air Force.

AREA NO. 9 - CITRUS AND SUBTROPICAL FRUITS PROCESSING AND PRODUCTS - SOUTHERN LABORATORY

Problem. The citrus and subtropical fruit production of the Southern Region is an expanding industry with the need for the development of better, as well as new-type consumer products, and for the improvement of present or invention of new processing procedures and machinery. These advances are required to regularly utilize the currently large production, particularly of oranges and grapefruit, and the anticipated higher production of these fruits, to the economic advantage of the growers and consumers. Basic research is needed to lay the groundwork for these advances. This research is needed, for example, on the composition and physical nature of essential oils, flavonoids, including bitter constituents, constituents responsible for oxidized off-flavors, carotenoids, and the like, which determine many of the sensory characteristics, and which affect product quality and stability. Other problems whose solutions are dependent upon the availability of more detailed compositional and physical data are: cloud stability, gelation, discoloration, fermentation, and the like. Increased production of citrus has stimulated the development of new products but many of these are urgently in need of improvement which will depend in part upon advances in basic research. New products are needed to attract new markets and also to reduce packaging and shipping costs. Research is needed to improve frozen citrus concentrates as processing procedures change, to develop better high density concentrate products, citrus powders, chilled juice and section products, pulp-fortified products, and to develop new or improved canned products which have a natural fruit flavor. Research is especially needed on grapefruit to develop practical methods for reducing the bitterness and harshness of juice products and to increase the use of grapefruit juice base in mixed fruit juice blends, drinks, concentrates and the like. Along with progress on product development there is a serious need to improve the actual processing procedures, processing equipment, and packaging operations and materials, to obtain and maintain the most desirable fruit characteristics. As an illustration, research is needed to develop less expensive dehydration equipment and an improved process for the production of citrus powders.

USDA PROGRAM

The Department has a continuing long-term program involving biochemists, organic chemists, food technologists, and a chemical engineer engaged in both basic and applied utilization research studies on citrus and subtropical fruits of the Southern Region to develop new or extended uses for these commodities.

Research to develop basic information on chemical composition and physical properties of citrus and subtropical fruits, and their products and byproducts is conducted at the U. S. Fruit and Vegetable Products

Laboratories at Weslaco, Texas and Winter Haven, Florida. This information provides the necessary basis for efficient research in developing new and improved food products and processing technology. At the Weslaco Laboratory the program includes investigations of the biochemical mechanism of the conversion of precursors to carotenoids in grapefruit as a basis for improvement of processing characteristics of and products from colored grapefruit. The Texas Agricultural Experiment Station (substation 15, Weslaco), Citrus Rootstock Investigations Laboratory (CR, ARS, Weslaco), and the Texas College of Arts and Industries are providing grapefruit of known history and conducting, or cooperating in conducting, on the tree tests. Additional research on chemical composition and physical properties is carried out under contract at the University of Oklahoma Research Institute, Norman, Oklahoma, on investigations of the effect of maturity of grapefruit on total flavonoids, naringin, and poncirin; and on the chemistry and nature of naringin and naringin-derived compounds to provide a scientific basis for the control of bitterness in processed grapefruit products. At the Winter Haven Laboratory the program includes investigations of the neutral fraction of orange peel extract with the aim of isolating, characterizing, and identifying those substances, particularly bitter principles, that are most detrimental to the flavor of orange products. Investigations are also in progress on the composition of essential citrus oils as related to flavor of juices, concentrates, powdered juice, and other products; on investigations of the chemical and physical nature of components of cloud of orange juice to provide better understanding and control of factors affecting stability of orange juice products; and on investigations of the identities, quantities and chemistry of components in Florida grapefruit responsible for excessive bitterness and harshness in processed products. Close consultation is maintained with the Florida Agricultural Experiment Station (Citrus Experiment Station, Lake Alfred) and the industry.

Research to develop new and improved food products is carried out at the U. S. Fruit and Vegetable Products Laboratories at Weslaco, Texas, and Winter Haven, Florida. At the Weslaco Laboratory the major applied effort is to develop products which will make greater and more efficient use of grapefruit. Emphasis at the present time is on the utilization of natural and debittered grapefruit juice and puree as bases for the development of improved fruit juice blends, drinks, and concentrates. This research is being carried out in part in cooperation with several state and private organizations. The cooperators provide fruit or raw materials, such as pulp and juice, of known history. Processing plant facilities are available from the Texsun Citrus Corporation (Weslaco) and Rio-Vac, Inc. (Harlingen). Formal agreements exist with the Texas Agricultural Experiment Station (College Station and Weslaco), with Texsun Citrus Corporation (Weslaco) and with Rio Farms, Inc. (Edcouch). Informal cooperation is maintained with Texas Citrus Mutual, Inc. (Weslaco), Texas Canners Association (Weslaco) and such other organizations as are found necessary for the procurement and processing of fruit. At the Winter Haven Laboratory research is in progress to develop high quality, "instant" citrus powders by new and improved processing technology as described below.

In the field of new and improved processing technology, research is being carried out at the U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida, to determine how the "foam-mat" type of air-drying can be applied for the preparation of dried citrus products of optimum flavor and stability. Foam-mat drying of orange juices, and grapefruit juices, is being studied. This research is conducted in cooperation with the Western Utilization Research and Development Division (ARS) and the Florida Citrus Commission under a formal memorandum of understanding.

The Federal in-house scientific effort at the Southern Division devoted to research in this area totals 20.4 professional man-years. Of this total 12.0 is devoted to chemical composition and physical properties, 3.0 to new and improved food products, and 5.4 to new and improved processing technology. The contract research involves an additional 1.4 man-years, all of the effort being on chemical composition and physical properties.

The following lines of work were terminated during the year: (1) Investigations on preservation of chilled citrus products to prevent spoilage and permit delivery of improved products; and (2) Investigations to develop new and improved processed products from selected minor fruits, with emphasis on avocados, limes and Meyer lemons (under new and improved food products).

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemical and Physical Properties of Flavoring Constituents of Florida Citrus and Subtropical Fruit Products. Basic work on the composition of essential citrus oils as related to flavor of citrus products was continued. Two new constituents of essential orange oil, delta-3-carene and rhodinal, have been identified. It has been found that during the process of determination of orange oil on silica gel columns isomerization of terpenes results. The possibilities are suggested that d-limonene is the precursor of alpha- and gamma-terpinene, terpinolene and p-cymene in orange oil; and that alpha-pinene is the precursor of camphene. The feasibility of low temperature isomerization of terpenes on silica gel might provide a commercial source for many of these isomeric products. The presence of hexanol, octanol, nonanol, decanol and undecanol in cold-pressed orange oil was firmly established. Two of these alcohols (nonanol and undecanol) are new constituents. Beta-sitosterol was isolated from orange cold-pressed peel oil. Three new terpenes (α-thujene, sabinene, and camphene), and a sesquiterpenoid having a conjugated ketone, have been isolated from grapefruit oil. The latter compound has the odor characteristic of grapefruit. The products obtained upon isomerization of limonene oxide have been identified as havenal, dihydrocarvone, and 1-acetyl-3-isopropenylcyclopentane. This may serve as a new commercial use for d-limonene. (S3 2-36).

2. Investigation of Bitter Principles and Flavonoids in Citrus Products. To date approximately 79% of the neutral fraction of orange peel juice extract has been accounted for in research on bitterness in oranges. This fraction is the largest and most bitter of those separated from this source. Tentative composition of the neutral fraction is: tangeretin, 4.9%; nobiletin, 35.5%; sinensetin, 24.2%; 3,5,6,7,8,3',4'-heptamethoxyflavone, 6.1%; and unidentified flavone, 8.6%. The structure of sinensetin has been definitely determined to be 3',4',5,6,7-pentamethoxyflavone and not the 3',4',5,6,8 as formerly thought. From the analysis of the neutral fraction it should be possible to determine which substances are responsible for most of its bitterness once the principal constituents are evaluated separately. A monthly examination of orange peel juice during the 1962-63 fruit season indicated a decrease in bitterness as the season progressed. Since the season was unusual due to the December freeze, the results will have to be checked in a normal year. If it can be shown that bitterness decreases late in the season to a point where it is no longer offensive, commercial producers of orange juice could increase their extractor pressures during this time without increasing bitterness in the product. (S3 2-37).

Contract research is being initiated at the University of Oklahoma Research Institute to investigate the changes which occur as grapefruit matures--in total flavonoids, poncirin, naringin, and naringin-derived compounds responsible for bitterness--to provide a scientific basis for the development of control methods and blending procedures in order to process uniformly flavored grapefruit products throughout the processing season. It is planned to study Texas grapefruit, at least in the initial investigations. (S3 2-39 (C)).

3. Factors Affecting the Physical Characteristics of Processed Citrus Products. A stable cloud is considered to be an essential characteristic of high quality orange juice products. In further work on the composition of cloud of orange juice, centrifugally sedimented solids and cloud have been recovered from orange juice at three stages of maturity and classified into three portions according to sedimentation rate. Suspended solids have also been recovered from pulp washings. All of the cloud fractions have been extracted with alcohol, acetone, and ether for the separation of lipids and water-insoluble suspended matter from the soluble solids of the juice. Ash, phosphorus, and nitrogen content of the insoluble solids have been determined. The suspended solids recovered by mild centrifugation (A) were found to have very high pectinesterase activity, while the cloud recovered by the highest available centrifugal force (B and C) showed significantly higher activity on the dry weight basis. Observed differences in composition between the easily sedimented particles (fraction A) and fine cloud (fractions B and C) indicate that at least some of the stable cloud components are not products of mechanical degradation of rag and peel. Fraction A contained more debris from cellular structure, as evidenced by higher cellulosic content, than did fractions B and C. The cloud fractions contained more lipids, and the solvent-insoluble portions of these fractions were

richer in pectin and nitrogen, and slightly higher in ash, than fraction A. The principal effect on the composition of cloud caused by freezing weather was an increase in solvent-insoluble solids for both A and B fractions. The information being developed may have considerable value in assessing effects of processing variables on juice quality. (S3 2-38).

4. Basic Investigations of Carotenoids in Grapefruit. Basic research on carotenoid formation in grapefruit has continued. For further experiments to establish whether carotenoids are made sequentially or by parallel synthesis, a means of purifying zeta carotene and phytofluene has been sought, with no success. However, the study of biosynthetic relationship of carotenes has proceeded, basing results on phytoene, lycopene and β-carotene, which can be adequately purified and account for about 95% of tomato carotenes. Radioisotope studies (incorporation of $C^{14}O_2$ into carotenoids) with tomatoes (used because of their more rapid synthesis of carotenoids) have provided data which support the postulate of simultaneous synthesis of lycopene and carotene considerably better than of sequential synthesis.

Preliminary results have indicated that the age of fruit seems to contribute slightly more to the seasonal decline of lycopene in colored grapefruit than factors correlated with date. Since most frozen trees had too "sets" of fruit this year, research was initiated to attempt to eliminate the variable of the individual tree by following lycopene content in each set of fruit on the same tree. The analysis of two "sets" of fruit on the same tree has again verified both the effect of age of the fruit and season of the year in producing the maximum lycopene content of red grapefruit. The data obtained this year indicate that season of the year has more influence than was shown last year. Application of $C^{14}O_2$ to grapefruit on the tree seems to have established that fruit, per se, and possibly carpels and peel, can individually make carotenes from carbon dioxide. When fruit were exposed, the total amount of label in the peel was higher at 48 hours than at 30 days, but label in the carotenoids was greater at the longer time, indicating continuous synthesis and a very slow turnover of carotenoids. When leaves were exposed, the total label in both peel and carpels was greater at 30 days than at 48 hours. Label in the carotenoids followed the same pattern but was much more pronounced again indicating continuous synthesis and a very slow turnover. (S3 2-34; S3 2-34 (Rev.)).

B. New and Improved Food Products

1. Improved Chilled and Canned Citrus Products. Good progress was made in continued research on the preservation of chilled citrus products. Heat treatments (140° - 160° F.), preservatives (sorbate, benzoate), and a storage temperature of 30° F. have been found to effectively increase the shelf life of chilled orange juice. Heat treatment and low storage temperatures reduce initial plate counts, inhibit microbial growth, and retard flavor change. Ascorbic acid in heat-treated, single strength orange juice was slightly more stable than that in unheated juice when the juices were stored at 0° F. At -90° F. storage, ascorbic acid was lost more rapidly

in the juice heated to 160° F. than in the unheated controls. Preservatives are beneficial under adverse storage conditions (above 30° F.) in minimizing changes due to microbial growth. Results from cloud determinations were inconclusive, but the trend was toward greater cloud stability with heat treatment. It has been confirmed that vitamin K_5 accelerates the loss of ascorbic acid in chilled orange juice. The vitamin appears to function as an oxygen carrier via a quinone-hydroquinone-type mechanism. This finding makes the use of vitamin K_5 as a preservative in chilled citrus products undesirable. (S3 2-35).

2. Development of New Grapefruit Based Beverages. Exploratory investigations were conducted on the use of debittered grapefruit juice in the preparation of fruit and berry flavored punches and drinks. The enzymatic debittering of grapefruit juice increased its flavor compatibility with such flavors as strawberry and raspberry. Debittering made it possible to increase the quantity of grapefruit juice in the juice blends and drink bases. Highly acceptable drinks were prepared with partially and completely debittered grapefruit juice blended with juice or puree from such fruits and berries as orange, strawberry, raspberry, blackberry, and plum. Taste panel testing has been used to determine preferences for levels of bitterness, acid and sugar in selected grapefruit juice-based drinks. The masking effect upon bitterness in grapefruit based drinks by blackberry puree was confirmed by a taste panel who compared the blackberry flavored drinks with other fruit flavored drinks. Seventy-three percent of the panel said blackberry flavored drinks were less bitter than plum flavored drinks; seventy-nine percent, less bitter than raspberry; eighty-seven percent, less bitter than strawberry; and ninety-three percent, less bitter than naranjilla flavored drinks. Storage studies were initiated on a four-fold (3+1) strawberry flavored grapefruit drink concentrate in plain and enameled cans at storage temperatures of 0° F. (control), 40° F., 50° F., 68° F., and room temperature. Initial results (1 month storage) showed that for the 68° F. and room temperature storage, samples from the enameled cans rated higher in flavor. Samples stored in plain tin showed a greater color loss than samples in enameled cans at storage temperatures of 40° F. and above. The use of resins such as Nylon 66 and Polyclar AT powders to absorb the principal bitter constituent(s) in grapefruit juice appears promising. Experiments demonstrated that these powders will absorb naringin from grapefruit juice without adversely affecting acidity, Brix, or vitamin C content. Trial runs with the powders and bitter grapefruit juice were successful. Both resins can be regenerated for reuse by washing with hot water or by extracting with alcohol. Control of bitterness by this means may have possibilities for industrial application. (S3 2-33; S3 2-40).

C. New and Improved Processing Technology

1. Application of Foam-mat Drying to Florida Citrus. Storage studies on orange juice powders produced by the "foam-mat" drying process have shown that storage at 85° F. accelerates the production of off flavors, compared to the 0° F. stored controls. Moderate storage life is possible at 70° F.

In investigations of factors affecting processing and product characteristics of foam-mat dried orange juice powder, none of the variables tested could be shown to increase the shelf life of the products in 85° F. storage. The addition of peel oil prior to foam preparation was found to reduce the rate of moisture removal during drying. The addition of SO_2 at 200 ppm in foam preparation did not prove advantageous. Likewise, further tests with nitrogen both in foam preparation and product packaging failed to show any differences in flavor stability between air and nitrogen processing and packaging. Processing of concentrates containing different amounts of pulp has shown that the rate of moisture removal during drying decreased as pulp content increased. Improvements in equipment and refinements of techniques in the foam-mat process have been made, leading to better initial products and confidence in the experimental results. (S3 2-32).

New research has been initiated to investigate foam-mat drying of concentrated grapefruit juices to provide a new grapefruit product of optimum flavor and high stability. The research will be cooperative with the Florida Citrus Commission, Florida Citrus Mutual, and WU. Preliminary experiments only have been conducted which indicate the feasibility of foam-mat drying concentrated grapefruit juices. Next phases of work will consist of investigation of time-temperature variables in the process, and freeze-drying of some concentrate to obtain grapefruit powder samples for comparative evaluation of quality characteristics and for control purposes. (S3 2-41).

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical Composition and Physical Properties

Lime, Bruce J. and Tucker, Donald M. 1962. Seasonal variation in Texas Hamlin and Mars orange juice, 1961-62. J. Rio Grande Valley Hort. Soc. 16, pp. 78-82.

New and Improved Food Products

Kew, Theo. J. 1962. Cloud and flavor stability in relation to density of frozen concentrated orange juice. Proc. Florida State Hort. Soc. 75, pp. 342-349.

Kew, Theo. J. and Veldhuis, M. K. 1962. Stability of frozen concentrated citrus juices following adverse storage. Food Technol. 16, pp. 119-122.

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Veldhuis, M. K. and Kew, Theo J. 1962. Storage time and temperature affect frozen citrus juice quality. ASHRAE (Am. Soc. Heating Refrig. Air-cond. Engrs.) J. 4(12), pp. 43-46.

New and Improved Processing Technology

Bissett, O. W., Tatum, J. H., Wagner, C. J., Jr., and Veldhuis, M. K. (SURDD); Graham, R. P. and Morgan, A. I., Jr. (WURDD). 1963. Foam-mat dried orange juice. I. Time-temperature drying studies. Food Technol. 17, pp. 210-213.

Veldhuis, M. K., Tatum, J. H., Wagner, C. J., Jr., and Bissett, O. W. (SURDD); Graham, Robert P. and Morgan, Arthur I. (WURDD). 1962. Progress in foam-mat drying of orange juice. Transaction 1962 Citrus Engineering Conf. 8, pp. 71-79.

General

Swift, Lyle James. 1963. Float check valve for use with a water aspirator. Chemist Analyst 52, p 23.

AREA NO. 10 - VEGETABLES PROCESSING AND PRODUCTS - SOUTHERN LABORATORY

Problem. Although extensive progress has been made in recent years in developing stable, attractive, and convenient to use vegetable products, new and improved processed products must be developed and means of stabilizing perishable vegetables provided to minimize the adverse effects of seasonable surpluses and unfavorable markets, and to provide an adequate supply of good food for a growing population. Product quality needs to be improved and processing cost reduced through the adaptation and application of the latest technological developments and nutritional findings. For example, a major problem of the cucumber industry, since most of the crop is brine-cured, is to improve the curing process so that no loss occurs in the value of the cucumber during the brine-curing and storage process and the cost of processing is reduced. As another example, a precooked, dehydrated, sweetpotato product has been developed which has good shelf life, when sealed under an inert gas. It reconstitutes to a product having the characteristics of freshly cooked and pureed sweetpotatoes. Applied research on a pilot-plant scale is needed to obtain additional engineering and processing data applicable to commercial production. Basic research is needed to improve the quality and storageability of the product. There is a continuing need in the use of vegetables for processing to investigate the characteristics of the raw material as these characteristics are affected by climate, soil, cultural practices, breeding and the like. Celery, already an important flavoring ingredient, could become much more important if the factors and constituents responsible for the intensity, variableness, and stability of its flavor could be controlled in processing, and processed products of improved flavor and convenience could be developed. Many vegetables grown in the Southern Region differ in their chemical and physical characteristics from the same crops grown in the more temperate regions; and several vegetable crops are grown almost exclusively in the Southern Region. More utilization research is needed to complement the Federal and State production research programs and to provide cooperation in the form of composition and processing studies.

USDA PROGRAM

The Department has a continuing long-term program involving biochemists, organic chemists, microbiologists, food technologists, and chemical engineers engaged in both basic and applied utilization research studies on vegetables of the Southern Region to develop new or extended uses for these commodities.

Research to develop basic information on chemical composition and physical properties of vegetables, their products and byproducts, is conducted as a basis for efficient research in developing new and improved food products and processing technology. Emphasis at the present time is on investigations of the flavor and aroma components in natural and pure culture

fermented cucumber pickle products, carried out at the U. S. Food Fermentation Laboratory, Raleigh, North Carolina, to provide the basis for producing pickle products of greater consumer acceptability. The North Carolina and Michigan Agricultural Experiment Stations, and the National Pickle Packers Association, cooperate in this research.

In the field of new and improved food products by processing of vegetables, research is being carried out at New Orleans, Louisiana, to improve or modify certain characteristics of the precooked dehydrated sweetpotato flake product with special emphasis on improvement of product acceptance and maintenance of quality during storage. Close cooperation is maintained with the Louisiana Agricultural Experiment Station, which furnishes sweetpotatoes of known history, and industry and industry associations. Research is in progress at the U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida, on the development of processed celery products of improved flavor and convenience. Research is also being conducted at the U. S. Fruit and Vegetable Products Laboratory, Weslaco, Texas, to develop new and improved processed products from southern grown vegetables other than sweetpotatoes and celery. The Texas Agricultural Experiment Station and industry associations provide raw materials of known history for this research.

Research on new and improved processing technology is conducted at New Orleans, Louisiana and at the U. S. Food Fermentation Laboratory, Raleigh, North Carolina. Pilot-plant investigations are being carried out at New Orleans on the production of a precooked dehydrated sweetpotato flake product to obtain engineering and other processing data applicable to commercial production. Processing variables being investigated include the effect of variety, curing, preheating, type of cooking, food additives and type of packaging. This work is closely related to the work of the Eastern Utilization Research and Development Division to improve the quality of processed potato products. The Marketing Economics Division, ERS (under a formal memorandum of understanding with the Southern Division, the Louisiana Sweet Potato Commission, the Louisiana State Agricultural Experiment Station, and the Louisiana State Department of Agriculture), conducts market tests on promising precooked dehydrated sweetpotato flake products to determine their consumer acceptance and market potential. In market tests to determine the type of package (can, glass jar or flexible pouch) that is best suited for retail market outlets for the flakes, Owens-Illinois Glass Co., Continental Can Co., and Lengsfield Bros. are cooperating by providing packaging materials. Milprint, Inc. and Continental Can Co. are cooperating with the Southern Division in packaging studies to determine the structure of a flexible package required for flakes for the retail market. At Raleigh the objective of the research is to improve cucumber processing technology and the quality of the products. Current emphasis is on investigations of methods for the controlled fermentation of cucumbers by application of pure culture techniques to fermentation practices (including differential control of microbial species in natural fermentations by chemical and physical means) in order to reduce processing costs

and improve product characteristics. A hydrolytic enzyme inhibitor extracted from sericea forage is under investigation for the prevention of softening of cucumber brine-stock during the curing process. The North Carolina Agricultural Experiment Station is cooperating in the design and execution of experiments concerned with postharvest treatments to maintain or improve cucumber processing characteristics, and develop leads to improved methods of processing. The Michigan State University (Department of Microbiology) provides technical assistance in the controlled fermentation studies. The National Pickle Packers Association contributes support to the research and supplies raw material.

The Federal in-house scientific effort at the Southern Division devoted to research in this area totals 17.8 professional man-years. Of this total 2.1 is devoted to chemical composition and physical properties, 10.1 to new and improved food products, and 5.6 to new and improved processing technology.

The following line of work was terminated during the year: (1) Basic research on isolation and characterization of flavor and odor constituents of celery (under chemical composition and physical properties).

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Identification and Characterization of Flavor and Aroma Components of Pickle Products. Basic research on the flavor and aroma components in natural and pure culture fermented cucumber pickle products was recently initiated in cooperation with North Carolina Agricultural Experiment Station, Michigan Agricultural Experiment Station, and the National Pickle Packers Association. It is planned to isolate, identify, and characterize these components to provide the basis for producing pickle products of greater consumer acceptability. Reliable methods for head-space sampling of foods for gas analysis and for measuring flavor components are being studied. Pure culture fermentations of cucumbers have been prepared for subsequent studies on flavor composition. These included duplicate 48-oz. jar lots using 34 lactic cultures representing four species (L. plantarum, L. brevis, Ped. cerevisiae and Leuc. mesenteroides). (S3 5-21).

2. Identification and Characterization of Flavor Constituents of Celery to Improve Processed Products. In fundamental studies of the composition of celery flavor and odor, a total of fifty-three of the sixty chemical compounds detected in distillates from expressed celery juice have now been isolated and identified. The total concentration of these compounds in celery juice is of the order of 0.5 to 1 ppm. A method for rapid essence recovery and flavor evaluation by a colorimetric ester test is being developed. Results have significance in that (1) identification of the major flavor contributing compounds will allow objective laboratory assessment of the effects of various processing conditions on the quality of manufactured

products and (2) a chemical test for total flavor, when completed, will provide a simple quality control procedure for use in industrial laboratories. Limited exploratory studies conducted on development of improved dehydrated celery stalk products indicate that flavor loss and toughness are problems involved. (S3 5-17, S3 5-23).

B. New and Improved Food Products

1. New and Improved Canned and Dehydrated Sweetpotato Products. Completed experiments on Goldrush sweetpotato flakes have confirmed previous findings that although combinations of antioxidants and synergists, including butylated hydroxyanisole, butylated hydroxytoluene, Tenox VI, α-tocopherol, citric acid and sodium pyrophosphate, extend shelf life of flakes sealed in air for a short time (48 days maximum), these additives are not sufficiently effective for commercial application. In other experiments, it was found that flakes could be readily processed from Centennial sweetpotatoes boiled in water, but flake storage stability in nitrogen was unsatisfactory. Flakes made from the Nemagold variety had a distinctive flavor and kept well when packaged in nitrogen but were not rated highly flavorwise. Flakes made from white varieties of sweetpotatoes were bland and starchy, yet were storage-stable in that they developed no hay-like flavor when stored in air for over a year. Flake products made from Goldrush by cooking in boiling water or in atmospheric steam above boiling water were acceptable and stable in nitrogen. Both cooking methods allowed the incorporation of solids ordinarily leached out and lost in steam cooking. The incorporation of sucrose up to 5%, dry weight basis, in the puree was found to improve the flavor and texture of "starchy" flakes. Packaging of the sweetpotato product in glass jars was shown to have commercial possibilities, especially for the retail market. Flexible packages may also have some promise if consistently gas tight packages can be attained. The sweetpotato product packaged with an oxygen content of less than 2% was shown to have the greatest storage stability. Antioxidant and synergist effects were minor in comparison.

Application of commercial amylolytic enzyme preparations in the "artificial curing" of freshly harvested sweetpotatoes shows promise of practical industry value as it may permit coordination of flake processing with canning, increases flake density, and enhances processability. Experiments employing five different commercial amylolytic enzyme preparations for conversion of starch of cooked puree from freshly harvested sweetpotatoes have shown promise in utilizing uncured sweetpotatoes for flake production. Greatly improved processing characteristics resulted when optimum time, temperature, and concentration conditions characteristic of each particular enzyme were used. Flake samples stored in nitrogen were quite stable for three months and panel evaluations were favorable. They indicated that the flavor of these flakes was "different" from those prepared from cured sweetpotatoes, but the general characteristic taste was

retained. Some samples have developed a slightly rancid or oxidized flavor after 6 months' storage. (S3 5-19).

2. Development of Modified Processing Procedures for New Varieties of Texas Vegetables. In continued green bean variety evaluation investigations, cooperative with the Texas Agricultural Experiment Station, 6 varieties and 3 strains of green beans were evaluated for desirable canning characteristics. The percent fiber in the pods of Pearlgreen, Abunda, B-3125-2-3-1, B-3489, B-3125-X-5-2 and Corneli 14 were above the .15 percent fiber tolerance allowed by the U. S. Standards for Grades of Canned Green Beans and Canned Wax Beans. Bush Blue Lake, Topmost and White Seeded Tendercrop were rated higher than the other varieties and strains because of lower fiber content in the pods. The exceptionally high fiber contents found in this past season's evaluations were due in part to several days of dry, hot south winds prior to harvest. Experiments with Pearlgreen variety green beans grown using different fertilizer treatments indicated it is possible to increase total yield of usable pods without sacrificing any of the desirable canning qualities of the pods by selected fertilizer application.

Limited effort has been made to develop a precooked, dehydrated carrot flake similar to the precooked, dehydrated sweetpotato flake. The bulk density of the carrot flakes is presently about 1/3 that of sweetpotato flakes and constitutes a major problem. Different kinds of hydrocolloids and other food materials and additives are undergoing evaluation in attempts to increase the bulk density of carrot flakes. Two experimental packs of carrot flakes have been canned in air and in nitrogen for stability studies at room temperature. One pack has no additive; the other contains salt. The experiments are still in progress.

Further experiments were conducted on firming the tissue of canning tomatoes in order to reduce peeling loss and increase drained weight. Homestead variety tomatoes (some with skins intact and some with skins pierced) were blanched in a 0.5% calcium chloride solution and canned. There was an average seasonal increase in drained weight of 5% attributed to better penetration in tomatoes which had had their skins pierced prior to blanching. The average increase in wholeness percentage was also slightly higher. The tests should be repeated using a 2% calcium chloride concentration in the blanch. (S3 5-16).

Research was initiated to apply newly acquired information on the chemical constituents of celery flavor to the development of processed celery products of improved flavor and convenience. Compounds pertinent to the flavor of celery have been found to be: 3-isobutylidene-3a, 4-dihydrophthalide; 3-isovalidene-3a,4-dihydrophthalide; 3-isobutylidene phthalide; 3-isovalidene phthalide; cis-3-hexen-1-yl pyruvate; diacetyl. The problems in dehydrating celery have been more specifically and clearly delineated in exploratory experiments. Fiber toughness in dehydrated celery appears to be associated with incomplete rehydration due to the

inability of the water to reach the interior portions of the pieces. The puffing technique of the Eastern Division has been investigated as a means of producing a more porous product with improved rehydratability. Small scale experiments indicated the technique may be of value, but conditions must be very closely controlled. Indications are that fiber toughness may be less of a problem with Pascal variety celery than with the Utah variety used earlier. Contrary to earlier observations, it has been found that detectable off-flavors do in fact develop during dehydration, in addition to the loss of the original flavor-inducing constituents through volatilization. Tests are in process of development which will be useful in the control of celery products and processing procedures. (S3 5-23).

C. New and Improved Processing Technology

1. Processing Investigations to Improve Quality and Reduce Costs of Canned and Dehydrated Sweetpotato Products. In pilot-plant studies of the production of precooked, dehydrated sweetpotato flakes, processing conditions have been established for making satisfactory flakes from cured sweetpotatoes of the Oklamor and Nemagold varieties. Additions of sugar and Mylase-P enzyme were required. Storage tests on the flakes have been initiated. Further work will be necessary on Centennial variety to develop satisfactory processing operations and a product with an acceptable flavor. Experiments conducted to evaluate pilot-plant scale processing of uncured Goldrush sweetpotatoes using enzymes showed that, although "good" flakes were produced, they were not considered comparable in taste to those produced from cured sweetpotatoes, and bulk densities were lower.

A cooperative institutional market test (SU, ERS, and Universal Foods) on the sweetpotato flakes, conducted in New Orleans, Louisiana, and Cleveland, Ohio, showed a highly favorable reaction to this new product by personnel and customers of the restaurants and other types of institutional outlets. In another extensive evaluation (2,000 participants), about 80% of the people liked the flakes as well as or better than fresh sweetpotatoes, and 90% liked them as well as or better than canned sweetpotatoes.

Cooperative studies to determine the structure of a flexible package required for flakes for the retail market are underway with Milprint Inc. and Continental Can Co. Results to date are very encouraging. Also, cooperation with ERS has been initiated to determine the type of package - flexible pouch, glass jar or metal cans - that is best suited for retail market outlets. Currently, flakes are being distributed commercially only to institutional outlets, whereas a major percentage of the U. S. food expenditures are in the household retail market.

Costs of precooked, dehydrated sweetpotato flakes produced at annual rates of 4.2 million and 14.6 million pounds in a hypothetical plant for processing 54,000 pounds of raw sweetpotatoes per hour were determined to be 50.0 and 36.9 cents per pound, respectively, using sweetpotatoes costing one cent per pound. This information was forwarded to industry for their consideration of commercialization.

Two plants are commercially producing the flakes at the present time. Although the initial plant has more than doubled its production for the 1962-63 season, total output for both plants is still considered small for sweetpotato processing plants. Plans for installation of a third plant have been announced by a third company. (S3 5-19).

2. Processing Investigations to Improve Quality and Reduce Costs of Fermented and Other Vegetable Products. Excellent progress has been made in the research to improve cucumber processing technology and the quality of the products. Formal cooperation is maintained with the North Carolina and Michigan Agricultural Experiment Stations, and the National Pickle Packers Association in this research. Earlier laboratory findings that the hydrolytic enzyme inhibitor (freeze-dried extract of sericea forage leaves) effectively prevents softening of cucumbers during brine-curing have been verified in small-scale brining tests (50-gallon drums) conducted in the field. The lactic acid fermentation and subsequent curing process developed normally in the inhibitor-treated lots, and the brine-stock was rated by a panel of plant operators to be highly acceptable for commercial use. Excellent results were obtained in blocking the softening action of added hydrolytic enzymes originating either from cucumber blossoms or from manufactured sources. In other work, it was found that pelletized freeze-dried lactic cultures can be used as effectively as broth cultures in making pure culture fermented dill pickles. Evaluation of these "semi-commercial" scale run dills after 5 months' storage showed that, in general, they rated "good" to "excellent" for acceptability for commercial use. Both of these developments offer promise for the production of superior pickle products, and for improving processing technology and reducing overall operating cost. (S3 5-20).

Assistance was given the Arkansas Agricultural Experiment Station in the evaluation of some new cucumber lines for brine-stock purposes. The brined cucumbers were evaluated for firmness, bloater content (hollow stock), color, shape, texture and overall acceptability for commercial use. The data will be used in rating the performance of breeding material as compared to known control cucumbers. This type of data is essential if better varieties are to be furnished the major cucumber growing areas of the country. (S3 5-16, S3 5-22).

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical Composition and Physical Properties

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Gold, Harvey J. 1962. Studies in the dehydration of celery. Proc. Florida State Hort. Soc. 75, pp. 336-342.

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New and Improved Food Products

Molaison, L. J. and Spadaro, J. J. (SURDD); Roby, M. T. and Lee, Frances H. (QMF&CI) 1962. Dehydrated diced sweetpotatoes--A pilot-plant process and product evaluation. Food Technol. 16, pp. 101-104.

New and Improved Processing Technology

Deobald, Harold J. and McLemore, Taylor A. July 24, 1962. Process for preparing a precooked dehydrated sweetpotato product. U. S. Patent No. 3,046,145.

ARS No. 11 - NAVAL STORES PROCESSING AND PRODUCTS

Problem. New uses for turpentine, rosin and pine gum need to be developed through research to provide new industrial markets for current and anticipated production of gum naval stores. These gum naval stores products face serious competition for markets from research-developed products, especially those from the chemical and petroleum industries. As an illustration, turpentine has lost substantially all of its industrial solvent market to low-cost petroleum based solvents. New fundamental information about the chemistry, composition and properties of pine gum, rosin and turpentine is needed to fully exploit their unique characteristics in the production of new and improved industrial products having utility as industrial chemicals, polymers, plastics, elastomers, resins, plasticizers, surface coatings, textile finishes, odorants, insecticides and herbicides. There is also a serious need to improve existing processes and develop new processing technology for the industry.

USDA PROGRAM

The Department has a continuing long-term program at Olustee, Florida, involving organic chemists and a chemical engineer engaged in both basic and applied research to discover and develop new and improved uses for pine gum and its products. In basic research on the chemical composition and the properties of gum naval stores materials the emphasis is on the isolation and characterization of some of the unidentified components of pine gum, rosin, and derivatives to obtain information that will aid in the further industrial utilization of gum naval stores. The U. S. Forest Service cooperates by supplying selected samples of pine gum. Informal cooperation is maintained with industry. In research to develop new and improved industrial products from pine gum, rosin, turpentine, or their components, conversion of the resin acids derived from gum rosin and pine gum to new polyfunctional products by reaction with suitable chemicals is under investigation to develop intermediates for production of resins, plastics, plasticizers, and other products. Another research approach involves the condensation of the unsaturated (olefinic) materials present in pine gum with certain reactive chemicals (dienophiles) to produce industrially useful chemicals. Research is also being conducted to develop uses for photosensitized oxidized pine gum and components, primarily in the fields of plastics and rubber. Other research includes investigations to convert turpentine and rosin into polymerizable products suitable for making new polymers, plastics, and resin; to prepare chemical intermediates and modified rosin compositions by hypochlorite reaction of rosin and resin acids; to convert rosin, resin acids, and resin acid derivatives to polyfunctional compounds useful in plastics, resins, and surface coatings by formaldehyde addition and subsequent reactions; and to produce reactive chemical intermediates from turpentine by reaction with inexpensive low molecular weight compounds. The Pulp Chemicals Association supports a Fellowship at the Naval Stores Laboratory for the purpose of conducting research to develop a suitable method for determining rosin and rosin derivatives in protective coatings, a necessity if rosin is to be allowed in certain types of these coatings from which it is now excluded. Additional research is in progress under contract at the University of Cincinnati, Cincinnati, Ohio, on the application of the oxo and related reactions to terpenes and resin acids to produce new, useful alcohols, aldehydes, and/or acids, and the characterization of the products thus obtained.

Contract research at Cornell University on the synthesis of terpene alcohols and glycols for use in the production of new and useful terpene derived polymers is being initiated.

The Federal in-house scientific research effort in this area totals 15 professional man-years. Of this total 13.7 is devoted to new and improved industrial products and 1.3 to new and improved processing technology. The contract research involves an additional 1.5 man-years on new and improved industrial products.

The following lines of work were terminated during the year: (1) The isolation and characterization of some of the major unidentified components in pine gum, rosin and some of their derivatives (under chemical composition and physical properties), and (2) the photochemical addition of suitable reagents to resin acids of pine gum origin to produce new chemicals of potential utility in the fields of surface active agents, textiles, paper and plastics (under new and improved industrial products).

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Composition and Pnysical Properties of Pine Gum. Research is in progress to isolate and characterize some of the unidentified components of pine gum and its derivatives to provide basic information that will aid in the further industrial utilization of gum naval stores products.

The new resin acid recently isolated from slash pine gum has been termed elliotinoic acid. Upon reduction of this acid, the previously isolated elliotinol is produced. Elliotinoic acid is the first bicyclic diterpene acid to be isolated from the oleoresin of the pine. It is not present in any detectable quantities in longleaf oleoresin nor in wood rosin. The ultraviolet and infrared spectra of the new acid indicates that it has two conjugated double bonds and at least one exocyclic methylene group. The acid forms a crystalline methyl ester and sodium salt. Indications are that the methyl ester has a double bond arrangement similar to that proposed for a closely related acid isolated by other workers from the bark of the common juniper. Dehydrogenation of the methyl ester gave a compound with ultraviolet absorption characteristic of a trisubstituted naphthalene (S5 2-36).

The project under which this work was conducted has been discontinued and the personnel transferred to a project on development of polyester resins from pine gum derivatives. Since some of the components separated during the course of the research have considerable influence on polyester resins obtained from pine gum, further work will be done on some of these materials under the latter project.

B. New and Improved Industrial Products

1. Development of Intermediates for the Production of Resins, Plastics, and Plasticizers from Pine Gum and its Components.

The preparation of chemical intermediates from naval stores products for use in industrial applications has continued. Reductive amination of ethyl pinonate with aliphatic mono- and diamines was found to proceed satisfactorily, but aromatic amines like aniline and p-phenylenediamine did not react. By this simple reductive amination process, products useful as initiators and crosslinking agents for polyurethanes and epoxy resins should be obtainable.

A study of the reactions of pinonic acid and homoterpenylmethyl ketone with acetylene to produce derivatives having potential industrial value has been completed.

Procedures were developed for the catalytic conversion of α-pinene epoxide to α-campholene aldehyde with zinc bromide, and for the preparation of α-campholenyl-α-campholenate by the Tischenko reaction from the α-campholene aldehyde. The preparation of 2,2,3-trimethyl-1-vinyl-3-cyclopentene by pyrolysis of the acetate of α-campholenyl alcohol gave poor results; methyl octyl xanthate gave good yields of octene-1. Since presumably good copolymers have been prepared in contract research at University of Arizona from ethylene propylene and the dimethyl-2, 7-octadienes from the pyrolysis of pinane, the preparation of vinyl olefins from α-campholene aldehyde and α-campholenol has more appeal since these should also copolymerize with ethylene and propylene. Research on the pyrolysis of α-campholenol will be continued. Esters of adipic, oleic and α-campholenic acids and α-campholenol will be prepared and oxidized (S5 2-38).

In another phase of work, the production of reactive chemical intermediates from turpentine by reaction with low molecular weight reagents is under investigation. Potentially useful new products have been made by photosensitized oxidation and by the reaction of dienophiles with terpenes. The major products of the photosensitized oxidation of limonene have been isolated and their physical properties determined; and progress has been made in identifying the photosensitized oxidation products of 3-menthene. Conjugated dienols and trienes obtained from limonene have unusual structures which should make them suitable as co-monomers in various types of polymers. The products from 3-menthene should include direct precursors of menthol. All available homoannular menthadienes were found to react with acrylonitrile. Further investigation of the reactions of terpenes with strong acid has revealed that the conjugated homoannular diene α-terpinene yields mixtures of gamma-terpinene, 2,4(8) menthadiene and itself similar to those obtained with other menthadienes. A combination of distillation and crystallization was used to separate the four isomers formed in the addition of acrylonitrile to α-terpinene. They were hydrogenated to saturated nitrile and amines. The two solid isomers yield the same saturated amine while the two liquid isomers yield a different one. The data obtained establishes that the two solid acrylonitrile-α-terpinene adducts are one exoendo pair while the two liquids are the other. The results to date under this project indicate that of the various reactions screened, the reaction of the terpenes with dienophiles offers the best possibilities for obtaining inexpensive useful chemical intermediates from turpentine. Emphasis of future work will be on the utilization of the primary products from the terpene - dienophile reactions, and on attempts to obtain good yields of simple addition products from the pinenes (S5 2-40).

2. Addition of Chemicals to Rosin Acids With Emphasis on Photochemical Methods to Produce Chemicals Useful in Manufacturing Surface Active Agents, Textiles, Paper and Plastics.

Studies were continued on the photochemical addition of reagents to resin acids. Palustric acid and levopimaric acid yielded dehydroabietic acid by treatment with visible light plus a molar amount of photosensitizer. Several other resin acids investigated did not react under similar conditions. The hydrogenation of levopimaric acid transannular peroxide was examined and conditions established for the selective reduction of the carbon-to-carbon double bond. Since this technique should yield a peroxide of greatly increased thermal stability, the findings will be applied to the hydrogenation of photosensitized oxidized pine gum to produce a product for testing as a vulcanizing agent for gum rubber, polyethylene, and

poly(propylene-ethylene) synthetic rubber. A scaled-up process for the preparation of photosensitized oxidized pine gum is under development. Photosensitized oxidized pine gum is a crude mixture of peroxides of known structure containing about 0.5-0.6 equivalents of peroxide per mole of resin acid persent. This product should be preparable for 10 to 15 cents per pound. A search for industrial uses for this product is underway, including its use as an intermediate in the manufacture of epoxy resins. Research will be continued on the thermal rearrangement of the acid peroxide and its ester, employing levopimaric acid transannular peroxide for the preliminary studies. (S5 2-37; S5 2-47).

3. Conversion of Turpentine and Rosin Acids into New Polymers, Protective Coatings and Resins. The resin acids of gum rosin and pine gum are monofunctional, i.e., they contain one carboxyl group. Conversion of these monofunctional substances to new polyfunction products by reaction with suitable chemicals is under investigation to develop intermediates for production of resins, plastics, plasticizers, and other industrial products.

In further research on hypochlorite modification of rosin and resin acids to produce intermediates for use in industrial resins, surface coatings, plastics, emulsifiers and similar materials, a pure, solid hydroxyabietic acid has been isolated in 30% yield from the reaction of sodium levopimarate with sodium hypochlorite. The conversion to an hydroxyabietic acid is unique. A number of useful compounds might be prepared from a reaction of this type. By understanding the reactions of hypochlorite with pure resin acids, it will be possible to apply the results to the reaction with sodium hypochlorite. The conversion to a hydroxyabietic acid is unique. A number of useful compounds might be prepared from a reaction of this type. By understanding the reactions of hypochlorite with pure resin acids, it will be possible to apply the results to the reaction with rosin. (S2 5-44).

Research has continued on the preparation of polyfunctional compounds from rosin, resin acids and resin acid derivatives by formaldehyde addition and subsequent reactions, for use in plastic formulations, resins and surface coatings. Industrial concerns have expressed interest in the methylolated rosin obtained by addition of formaldehyde to rosin, and in the glycol produced by reduction of the carboxyl group of the methylolated product. Use of neutrals-free rosin in place of rosin has been found to give more homogeneous end products. An analytical system has been developed for determination of the major components in the neutrals-free resin acid mixtures and is being applied in obtaining a better understanding of the methylolation process.

Further research on the preparation of these polyfunctional compounds through reaction with formaldehyde has shown combined dehydro- and dihydroabietic acid in neutrals-free resin acids in sufficient quantity to account for the portion of the resin acids not reacting with formaldehyde. The reaction of pure levopimaric acid with formaldehyde gave a crystalline product. The diol obtained on reduction of the carboxylic acid function is also a crystalline solid. Pure abietic acid has been reacted with formaldehyde in a closed reactor, but the products are as yet ill-defined and may be mixtures of mono- and dimethylolated materials. Although the methylolated materials obtained from pure levopimaric acid and pure abietic acid may never attain commercial importance, studies of these pure materials make possible a better understanding of the reactions involved in the methylolation of rosin or rosin derivatives. Products of the latter reactions do have much commercial promise and have stirred considerable interest in industry. The large scale methylolation of neutrals-free resin acids will be carried out, the products esterified, and the reduction to diols investigated. The structure of the methylolation products from pure resin acids will be investigated. (S5 2-43).

Further research to prepare and evaluate improved polyester resins from pine gum derivatives has led to the establishment of optimum conditions and concentrations for preparing the β-propiolactone and acrylic acid adducts of resin acids. An improved method has also been developed for separating these adducts from the modified rosin. A series of unsaturated polyesters prepared from β-propiolactone-modified rosins is undergoing evaluation by a commercial producer of polyester resins. A practical technique has been developed for the preparation of low acid number polyester resins from both fumaric- and β-propiolactone-modified rosin. The two-stage procedure avoids most of the danger of jelling the polyester and gives high molecular weight esters. Work was completed on the preparation of unsaturated polyesters from gum rosin modified at several levels with β-propiolactone; each level was condensed with the glycol ester of fumaric acid. Copolymers of these materials with styrene were evaluated and found to have commercially acceptable properties. Elliotinoic acid, the first bicyclic diterpene acid isolated from the oleoresin of the pine, was characterized. The presence of elliotinoic acid, elliotinol, and other minor constituents in American pine gum probably accounts for its unique resistance to crystallization. Evaluation of esters and polyesters of pine gum derivatives will be continued. Some of the components of rosin and modified rosin that influence the properties of polyester resins prepared from it will be isolated and characterized. Attempts will be made to develop a practical method for separating the dibasic β-propiolactone adduct in relatively pure form from the unreacted resin acids. (S5 2-42).

Progress has been made in contract research at the University of Arizona on preparation and evaluation of a number of polymers from pine gum derivatives. It has been found that vinyl pinolate will bulk polymerize, even without removal of its copper resinate stabilizer, to give conversions as high as 65% and inherent viscosities between 0.2 and 0.3. Conversions as high as 94% are obtained in emulsion polymerization. Copolymers of vinyl pinolate and vinyl chloride or vinyl acetate in benzene solution were prepared in about 45% conversion, with inherent viscosities of 0.16. Attempts to dehydrate polyvinyl pinolate by heat in order to reduce the number of hydroxyl groups led only to crosslinked insoluble products. The polymerization experiments with vinyl pinolate will be continued. Research will be initiated on the copolymerization of terpenes with olefins such as ethylene and propylene. (S4 1-89(C)).

A large concentration of rosin is undesirable in certain types of surface coating vehicles and Federal Specifications TTR 266 now specifically exclude rosin derivatives. If there were a satisfactory procedure for determining the rosin content, specifications could probably be modified to permit the use of small quantities of rosin in the protective coatings. Research in cooperation with the Pulp Chemicals Association has resulted in the development of a method for the determination of free rosin acids in maleic modified alkyd vehicles. Preliminary results indicate that the method can be extended to the determination of total rosin derivatives in such products. It is essential that such a method be developed if rosin is to be allowed in certain types of protective coating formulations from which it is now excluded. (S5 2-39).

C. New and Improved Processing Technology

Processing Investigations to Produce Naval Stores Products of Improved Quality at Lower Costs. In continued research to develop an improved process for isolation of pure levopimaric acid from pine gum, the yields of a 95% levopimaric acid product obtained by a three-stage 2-2mino-2-methyl-propanol process (precipitation

and two recrystallizations), using recycling, were 75% for longleaf gum (containing 38% levopimaric acid in its resin acids) and 31% for slash gum (containing 27% levopimaric acid in its resin acids). The equivalents of amine employed per equivalent of levopimaric acid for the two types of gum were 1.6 and 1.2, respectively. Although the low yield obtained from slash gum will make it desirable to use longleaf gum which has a higher initial levopimaric acid content, the process developed is simple and should be attractive commercially. Several amines other than the aminopropanol and solvent other than acetone were investigated for use in the precipitation step of the process without success. Current research has been concerned with the development of a method of analysis for the mixtures of resin acids encountered in the isolation of levopimaric acid by amine precipitation from acetone. Only levopimaric acid is readily determined at present. To ascertain the abietic type acids present use was made of gas-liquid chromatography and ultraviolet spectroscopy to determine the change in composition resulting from acid isomerization. At present, the data from gas-liquid chromatography indicate greater percentages of resin acids than the ultraviolet data. Work will continue along the same line with emphasis on developing a method which will give correlation between gas-liquid chromatography and ultraviolet spectroscopy. (S5 2-41).

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New and Improved Industrial Products

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New and Improved Processing Technology

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AREA NO. 12 - PROCESSING AND PRODUCTS - SUGARCANE

Problem. Quotas established by the Sugar Act effectively prevent the accumulation of surpluses by limiting production to estimated requirements at stable, and normally low prices for sugar. Prices received by farmers of the United States and Puerto Rico for sugarcane are based upon the recoverable sugar content of the cane; and the rising costs of production and processing make imperative the more efficient recovery of increased amounts of sugar to provide adequate returns for both processors and growers. Currently recovery of 75% of the total sugar in the cane is considered satisfactory in Louisiana, and about 83% in Puerto Rico and Hawaii. Improved processing methods could increase the recoverable sugar to at least 85% in Louisiana and over 90% in other areas. The development of more efficient processing methods depends in turn upon the acquisition of adequate data on the quantitative composition of juices extracted from sugarcane, and of materials processed to recover sugar. The chemical industry provides a promising potential for the utilization of additional sugar since more than 40 billion pounds of chemical products are produced annually and sold to every section of American industry. More information is needed on the chemistry and properties of products from sugar to expand their utilization and on the application of these derivatives in the production of plastics, protective coatings, emulsifiers, detergents and the like.

USDA PROGRAM

The Department has a continuing long-term program involving at the Southern Utilization Research and Development Division organic chemists and chemical engineers engaged in basic research on the composition and properties of sugarcane, sugarcane juices and derived products, and in applied research directed to the development of new and improved sugarcane processing technology.

Basic and exploratory studies are being carried out at New Orleans, Louisiana, on the composition of sugarcane and sugarcane juices as a basis for developing more efficient methods for economical production of high grade end products. Materials used in this research are being obtained from cane processed for pilot-plant experiments in cooperation with the American Sugar Cane League.

Research on new and improved processing technology is being conducted at New Orleans, Louisiana, the U. S. Sugarcane Products Laboratory, Houma, La., and the Audubon Sugar Factory (Louisiana State University), Baton Rouge, Louisiana, to develop on a pilot-plant scale novel and more effective means of clarifying sugarcane juice, and improved methods of processing and purifying sirups to obtain greater recovery of raw sugar of higher quality at lower costs. This research is planned and conducted in close cooperation

with the American Sugar Cane League and individual sugar companies. Sugarcane for the work is furnished by the League and use of the Audubon Factory for milling of the cane through the cooperation of Louisiana State University. Cooperation is also maintained with the Crops Research Division, ARS (U. S. Sugarcane Field Station, Houma, La.). Informal cooperation is maintained with the industry in evaluating quality of raw sugar and economic aspects of new processing methods.

Other research on chemical composition and properties has been initiated under a grant of P. L. 480 funds to Kyoto University, Kyoto, Japan, for isolation and identification of the nucleic acid derivatives of cane molasses, in order to obtain information applicable to expanding the utilization of molasses industrially and in feeds (project duration - 2 yrs.).

The Federal in-house scientific effort at the Southern Division devoted to research in this area totals 10.8 professional man-years. Of this total 3.7 are devoted to chemical composition and physical properties and 7.1 to new and improved processing technology. P. L. 480 research involves 1 grant for research on chemical composition and physical properties.

During the year emphasis on pilot-plant development of new and improved methods of clarification was reduced somewhat to permit initiation of promising research to investigate the qualities of clarified sugarcane juices in relation to the refining quality of raw sugars.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Basic Studies of the Chemical Composition and Physical Properties of Sugarcane Juice and Its Products. Basic studies were continued to gain more complete knowledge of the composition and properties of sugarcane juice and its products. Further study of the two water-soluble polysaccharide fractions of the gums present in juice solids and molasses showed that alcohol precipitation effects the separation of fraction A, with very little occlusion of fraction B which is obtainable by adsorption on carbon. The alcohol-precipitable gum (fraction A) from juice solids contained 37.5% glucose, 6.2% fructose, and had a specific rotation, α,of + 67°. This gum could be separated into two sub-fractions by alcohol precipitation--one having α + 161° and 7.1% fructose; the other, α + 133° and 19.2% fructose. Starch was not present in these materials. The non-alcohol precipitable gum (fraction B), which was separated and determined on carbon-Celite columns, contained 40.2% glucose and 14.0% fructose. The fact that fructose is a major constituent of the various fractions of water-soluble sugarcane gum is an important new finding.

Methods were developed for determining pentose content, in addition to the other component sugars, in the gum fractions of juices, sirups and molasses. Routine procedures have also been devised for the separation and estimation

of starch and gum fractions A and B in sugarcane products. In connection with the starch work, sugarcane starch was isolated and found to give the same iodine color yield as Baker's soluble starch used as a standard in colorimetric starch determinations. In completed work on the determination of the amounts of the trisaccharide, kestose, in juices, concentrations approximating 0.3% were found in three samples of lyophilized juice solids. The concentration of mannose in a sample of Florida molasses was determined in cooperative work with the New York Sugar Trade Laboratory.

Knowledge of the chemical and physical properties of the minor constituents of sugarcane will aid in the pilot plant studies on new methods of clarification. Methods for the quantitative determination of these constituents are essential in order to determine the effectiveness of various clarifying agents and procedures. The measurement of the physical properties such as the specific rotation of the polysaccharides from the various stages of processing may indicate whether bacterial polysaccharides are formed. The formation of such polysaccharides in addition to the natural polysaccharides already present would represent a loss of sugar as well as an interference in subsequent steps in processing and refining. (S5 1-71).

B. New and Improved Processing Technology

1. Improved Processing Procedures for Clarifying Sugarcane Juice. Experiments were continued to improve methods of clarification of sugarcane juice. A definite relationship has been established between phosphate content of mixed juice and its response to lime clarification over a wide phosphate range. Clarity, filterability, and clarification efficiency values increased correspondingly with phosphate, reaching a maximum at 0.6 pounds of phosphate (as P_2O_5) per ton of cane processed. Clarification quality gradually deteriorated as the phosphate content increased beyond this optimum value. Phosphate content of mixed juice could become an important factor in cane variety and selection and in promoting selective harvesting and cane blending at the mill to insure maximum juice clarification efficiency and the production of high quality raw sugar. Limiting the variations in phosphate concentration would also insure a more uniform mud rate to the filters, and allow maximum capacity factory operation during a higher percentage of time.

Two cationic and one anionic flocculant were investigated in further experiments to improve the clarification of sugarcane juice. The anionic, Separan AP-30, continued to yield greater improvement in settling. Best results were obtained with the combination of Separan and bentonite in clarifying juices from clean cane. Limited tests were made to explore possibilities of magnesia and starch, with negative results.

In cooperative work with Southdown, Inc., a novel test procedure (Millipore filtration test) has been found to give good correlation of the filterability of sirups with that of raw sugars produced from them, but no significant relationship of clarified juice filterability to sugar quality. The

The Millipore test of sirups and sugars at average juice density, 15% solids, is more sensitive than the standard pressure filtration of high density refinery sirups, and showed a relationship to refinery experience in processing the raw sugars which the standard test failed to reveal. A large commercial refiner is extremely interested in the possibilities of the novel test for evaluating raw sugars.

Standard pilot-plant processing experiments to evaluate four new canes were conducted during the 1962 harvest season. The data obtained will be used by the American Sugar Cane League in evaluating processing characteristics of the commercial and unreleased canes. (S5 1-70).

2. Composition of Sugarcane in Relation to Processing. Compositional work on juices from freeze damaged canes (1960 and 1961 seasons) has been completed. The data on the non-sugars in such juices which affect processing quality should be helpful in devising the most efficient means for processing freeze damaged canes.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical Composition and Physical Properties

Friloux, James J. and Cashen, Norton A. 1962. The isolation, separation, and identification of the principal phospholipides of sugar cane juice. J. Agr. Food Chem. 10, pp. 509-511

Martin, L. F. 1962. Complex organic nonsugars in the refining process. Proc. Tech. Sess. Bone Char, 7(1961), pp. 3-19

Roberts, E. J., Jackson, J. T., Fort, C. A., and Martin, L. F. 1962. Separate determination of glucose and fructose and estimation of other reducing substances in molasses. Intern. Sugar J. 64, pp. 197-201

New and Improved Processing Technology

Coll, E. E. (SURDD); Davidson, L. G. (Crops Research Div., ARS); Stewart, C. W. (Audubon Sugar Factory, LSU); and Guilbeau, W. F. (SURDD). 1962. Milling and processing qualities of cane combined in short pieces compared with whole-stalk burned cane. Sugar Bull. 40, pp. 212-219

Coll, E. E. Guilbeau, W. F., Fort, C. A., and Jackson, J. T. 1963. Comparative pilot-plant tests on a few approaches to sugarcane juice clarification. Sugar Bull. 41, pp. 110-115

Guilbeau, W. F., Coll, E. E., and Jackson, J. T. 1962. Sugarcane mud filtration. Sugar Bull. 41, pp. 62-66. Republished: Intern. Sugar J. 65, pp. 141-143 (1963)

Smith, B. A. (SURDD); Sanchez-Nieva, F., Vasquez-Romero, R., and Carlo-Velez, L. A. 1963. Production of liquid sugars. Ion exchange adapted to confectionery in Puerto Rico. Sugar y Azucar 58(4), pp. 33-36

General

Jones, Marie A. 1963. Sugarcane and sugarcane products - A bibliography of research at the Southern Utilization Research and Development Division of the Agricultural Research Service, U. S. Department of Agriculture. U. S. Dept. Agr., Agr. Res. Service, Sou. Util. Res. and Dev. Div., 55 pp. (Mimeographed)

AREA NO. 13 - REPLACEMENT CROPS - UTILIZATION POTENTIAL

Problem. Farmers could achieve more economic use of their land if new and profitable crops were available for their choice that would have different end-use patterns from those presently grown. For example, it would be advantageous to develop a new oilseed crop yielding unique fatty acids that could find industrial use in applications for which acids from presently available domestic oilseed crops are unsuitable. To develop a new crop, three basic steps are involved: (1) Survey of wild plants, in cooperation with plant scientists, to identify those having both potentially valuable components and promising agronomic potential for use in the U. S.; (2) detailed physical and chemical characterization of components of interest to obtain clues to likely end uses; (3) selection of the most promising species followed by additional utilization research to explore uses and demonstrate industrial potential and by additional agronomic research to establish proper cultural practices and select the best strains and varieties. Only after these steps have been successfully accomplished can a proposed new crop be offered to agriculture and industry for introduction and development. Obviously, a program of this type is a long-range one. Yet, whether the future of agriculture involves conditions of surplus, of greater emphasis on foods and feeds, or of necessity for greater national self-sufficiency, the nation will benefit from availability of optimum, practical crop plants to serve its needs.

To achieve the objective, survey and characterization work needs to be greatly increased, since the greater the number of species examined, the greater will be the opportunities for finding plants meeting the criteria of high utilization and agronomic potential. Work of the Department has already revealed several promising sources of new potentially valuable water-soluble gums, pulp fibers, and oils containing unique fatty acids such as hydroxy unsaturated acids, capric acid, epoxidized acids and unusual long-chain fatty acids. In order to demonstrate the potential of these new materials, further work is required on their physical and chemical properties and reactions, on processing to obtain maximum recovery from source plants and on byproducts from processing, such as oilseed meals.

USDA PROGRAM

The Department has a continuing long-term program involving organic and analytical chemists engaged at New Orleans, Louisiana, in research to develop and evaluate industrial chemical products from the oils of certain new oilseed crops having production potentials as replacement crops. Oils from the seeds of the plants Limnanthes and Cuphea, rich in unusual long-chain unsaturated acids and capric acid, respectively, and from seeds of Umbelliferae such as parsley, carrots, fennel, dill, and coriander containing high percentages of petroselinic acid, are currently being investigated. The research is concerned with chemical modification of the oils and their

fatty acids to produce materials having potential utility in plastics, plasticizers, synthetic rubbers, protective coatings, and other industrial products.

Close cooperation is maintained with the New Crops Research Branch, Crops Research Division, in the procurement of seed and in joint evaluation of the potential of the new crops. The Pharmacology Laboratory of the Western Division, Albany, California, performs tests as needed to determine the physiological properties of the oils, their derivatives, and the meals. Louisiana State University cooperates by testing some of the chemical derivatives for antimicrobial activity. Other appropriate agencies in the Department of Agriculture and the State Agricultural Experiment Stations cooperate by evaluating the utility of some of the new compounds prepared from the oils. Informal cooperation is also maintained with industrial firms for evaluations of promising materials developed in the research.

The Federal scientific effort at the Southern Division devoted to research in this area totals 6.0 professional man-years. All of this effort is on industrial utilization.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Industrial Utilization

1. Industrial Products from Oilseeds Containing Capric and Unusual Long-Chain Unsaturated Acids. Investigations have continued on the chemical modification of new crop oils, and their fatty acids, from the seeds of the plants Limnanthes, Cuphea, and Umbelliferae to produce materials having utility in plastics, plasticizers, synthetic rubbers, protective coatings, and other industrial products.

Lactonization of L. douglasii fatty acids using perchloric acid catalyst was found to proceed satisfactorily, and other less potentially hazardous compounds are being screened as catalysts for the reaction. Gamma lactones produced in this manner should be interesting new chemical intermediates. Of a large number of additional compounds screened as catalysts for the lactonization of eicosenoic acid (a major fatty acid of L. douglasii), p-toluenesulfonic acid was the best although not as effective as perchloric acid. Fractional distillation of the methyl esters from L. douglasii oil yielded practically pure methyl eicosenoate. The distillation was accompanied by some double bond isomerization, resulting in the presence of isomers in the distilled methyl eicosenoate, but these isomers will not interfere with the intended preparation of the gamma-lactone from eicosenoic acid.

Screening of a number of chemical derivatives of capric acid (an important constituent of Cuphea seed oil) and other medium chain length acids showed that 4-(2-octenoyl)morpholide, 4-(2-nonenoyl)morpholide, 4-(2-decenoyl) morpholide, 4-(2-bromodecanoyl)morpholide, propargyl 2-bromodecanoate, and

2-(dodecenoyl)morpholide had good antimicrobial activity. These compounds have been submitted to the National Institutes of Health for screening as anticancer drugs.

In research on petroselinic acid (a major acid of Umbelliferae seed oil), methods for preparation of petroselinonitrile and petroselinamide have been improved. Reduction of the latter to petroselinylamine, reaction with acrylonitrile, and further reduction produced petroselinylamine-N-propylamine ("duomeen" petroselinate) in good yields. The "duomeen" compound may have potential as a corrosion inhibitor. Attempts are being made to form trimethylol compounds from aldehydes obtained by ozonization and reduction of petroselinic acid. These compounds should offer good possibilities for potential new uses.

Since fennel is one of the more promising crops for production of petroselinic acid, compositional information on samples of fennel seed and oil from Crops Research Division is being obtained to guide their research on development of this crop. Oil of fennel seed from different planting dates and spacings was found to average better than 70% in petroselinic acid content. Planting dates particularly affected oil content. (S5 5-45).

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Industrial Utilization

Dupuy, Harold P., Goldblatt, Leo A., and Magne, Frank C. September 4, 1962. 4-(12-beta-Cyanoethylricinoleoyl) derivatives of morpholine. U. S. Patent No. 3,052,680.

Dupuy, Harold P., Goldblatt, Leo A., and Magne, Frank C. January 1, 1963. 1,12-di-beta-Cyanoethoxy-9-octadecene and method of preparation. U. S. Patent No. 3,071,611.

Dupuy, Harold P., Goldblatt, Leo A., and Magne, Frank C. February 26, 1963. 4-(12-Acetoxyricinoleoyl) derivatives of morpholine and method of preparation. U. S. Patent No. 3,079,387.

Dupuy, Harold P., Goldblatt, Leo A., and Magne, Frank C. February 26, 1963. 4-(Ricinoleoyl) derivatives of morpholine. U. S. Patent No. 2,079,388.

Fore, Sara P., Ward, T. L., and Dollear, F. G. 1963. The preparation of lauryl alcohol and 6-hydroxycaproic acid from petroselinic acid. J. Am. Oil Chemists' Soc. 40, pp. 30-33.

Holmes, R. L., Moreau, J. P., and O'Connor, R. T. 1962. Preparation of dodecylamine and 6-aminohexanoic acid from petroselinic acid. J. Am. Oil Chemists' Soc. 39, pp. 411-414.

Marvel, C. S. and Dykstra, T. K. (Noyes Chemical Laboratory, Univ. of Ill.); and Magne, F. C. 1962. Some polymers and copolymers of vinyl ketostearates. J. Polymer Sci. 62, pp. 369-377.

Placek, Lida L. and Dollear, F. G. 1962. The preparation and properties of some nitrogen-containing derivatives of petroselinic acid. J. Am. Oil Chemists' Soc. 39, 347-350.

AREA NO. 14 - RICE PROCESSING AND PRODUCTS

Problem. The productive capacity of U. S. rice growers has increased faster than domestic and export consumption over the past decade, thus limiting the income potentially available from this major world food grain. Detailed knowledge of chemical composition and physical properties, as related to processing is needed to guide milling, processing and product development of U. S. rices so that they can better meet the quality and new product requirements needed for expanded markets. New and diverse food products from rice that are easy to prepare, have flavor and texture appeal, and are economical to manufacture, are needed to increase the total consumption of rice both domestically and abroad.

USDA PROGRAM

The Department has a continuing long-term program involving at New Orleans, Louisiana, biochemists and analytical chemists engaged in basic and exploratory studies on the chemical and physical changes undergone by rice constituents during aging of rice after harvest, which will account for the improvement in cooking characteristics observed after storage of milled rice for a few months. Present research involves investigations of the biochemical characteristics of rice as affected by and in relation to aging and processing characteristics, with special emphasis on the susceptibility of rice starch to amylolytic action.

Close cooperation is maintained, under formal memoranda of understanding, with the Louisiana, Arkansas and Texas Rice Experiment Stations, who supply rice samples of known variety and cultural history for the experimental studies. The Rice Inspection Service, Grain Division, AMS, New Orleans, Louisiana, cooperates by providing assistance in milling and grading the rice samples used in the research investigations.

The Federal scientific effort at the Southern Division devoted to research in this area totals 3.7 professional man-years. All of the present effort is on chemical composition and physical properties.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Studies of Chemical and Physical Changes in Rice as Affected by Aging and Processing. Recent research on rice has been directed toward basic investigations of the biochemical characteristics of rice as affected by and in relation to age and processing characteristics. Fundamental information of this type is essential for efficient development of practical processes and products so as to increase the utilization of this grain.

Studies of the chemical and physical changes accompanying the aging of 1962-crop Bluebonnet-50 and Nato varieties of rough rice stored at both ambient temperature (77° F.) and 40° F. were completed. In investigations of the native alpha- and beta-amylase activity of the rices and the susceptibility of the starch to introduced alpha- and beta-amylases, the two varieties closely paralleled each other in overall amylolytic susceptibility and native amylase activity. Native alpha-amylase activity of samples aged at either temperature condition remained relatively constant, whereas beta-amylase activity showed a decreasing trend. The rice starch became more susceptible to amylolytic action during the aging process.

After aging, both varieties gave decreased losses in total solids to treating water at 90° C. The viscometric characteristics of the rices progressively increased during the 10-month aging, the lower aging temperature yielding the lower values. Improvement in the organoleptic characteristics for the aged rices of both varieties paralleled the well-defined changes noted in hydration and viscometric properties. There were no significant changes in the gross chemical composition (starch, protein and sugars) of the aged rough rices over the 10-month period, confirming previous results.

Preliminary results on similar experiments conducted with a second year's (1962) crop of Bluebonnet-50 and Nato rices are generally confirming the earlier findings. The same overall pattern of activity of the native amylase enzymes upon aging of the rice samples was observed. After aging of the rice, the starch became more susceptible to the attack of introduced alpha- and beta-amylases. There have been no significant changes in gross chemical composition of the rough rices over the 6-month aging period. However, water uptake and loss of solids to cooking water have progressively decreased at about the same rate for both storage conditions; and organoleptic properties, particularly cohesiveness after cooking, have improved. These changes are paralleled by an increase in viscometric characteristics. These observations of the difference between fresh and aged rice suggest physico-chemical changes in the nature of the rice constituents, perhaps the starch, as evidenced by its progressive change in viscometric characteristics, affinity for water, and amylolytic susceptibility. Still unknown, however, is the causative agent(s) which brings about such changes.

Observations resulting from a study of the aging process and previous investigations of heat-vacuum treatments of rice have led to the development of a method of effecting changes in freshly-harvested rice which bear striking similarities to those found in aged rice. The method consists of heating rough or white milled rice of 12-13 per cent moisture content in a closed container at temperatures between 90°-110° C. for 3 to 8 hours under controlled conditions which prevent loss of moisture from the grains. Characteristics of the treated rice resemble those of rice which has been aged for a period of 10 months or longer. The product has the hydration properties and pasting characteristics of an untreated aged rice and cooks

to a white, dry, fluffy consistency.

Work is in progress on the compilation, review and analysis of domestic and foreign information on rice physical and chemical properties as recommended by the USDA Rice Research and Marketing Advisory Committee, and the Rice Technical Working Group. (S1 4-12).

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical Composition and Physical Properties

Hogan, Joseph T. 1962. Biochemical studies of rice at Southern Laboratory. Rice J. 65(7), pp. 65-67.

AREA NO. 15 - PEACH PROCESSING AND PRODUCTS

Problem. The peach industry in the Southeastern United States is dependent to a large extent on the fresh market. For example, in the South Atlantic States in 1960, 14,870,000 bushels of peaches were produced of which 11,865,000 bushels were sold on the fresh market; slightly over 2,000,000 bushels were processed. A peach processing industry is needed in the Southeastern States to provide a profitable market for more of the edible peaches which do not meet fresh market standards and to rapidly convert a higher proportion of the overall crop to stable forms. Climatic conditions which favor rapid deterioration of fresh peaches both on and off the tree, erratic ripening periods and markets, and short lived peach orchards, are other factors contributing to the need for more extensively integrated fresh market-processing operations. There are technical problems preventing the more rapid development of the peach processing industry in the Southeastern States which must be overcome. Many of the peach varieties grown in the southeast require a modification of processing procedures to make satisfactory standard-type products. Still other varieties will not make standard-type products and new food forms must be found for them. Recent rapid advances in food science and processing technology make it possible through research to develop both new and improved peach products. These are needed to bolster the economics of the South's peach industry, as well as to provide the superior qualities, and greater convenience in food products, which the consumer now demands.

USDA PROGRAM

The Department has product and processing investigations in progress under contract at the Georgia Agricultural Experiment Station, Experiment, Georgia. This contract covers research to develop optimum procedures for the production and preservation of puree and clear juice peach concentrates; to develop optimum procedures for the preparation and the handling under simulated commercial conditions of refrigerated fresh peach slices; to develop optimum procedures for canning Southeastern peaches; and to conduct experiments directed to the development of partially dehydrated pasteurized peach products. Evaluation of different varieties of peaches, and of different processing variables are phases of the investigations. This research involves 1.2 man-years, and is carried out with the support of the Area Redevelopment Authority of the Department of Commerce.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

Experiments completed in contract research at Georgia Agricultural Experiment Station on the first season's (1962) packs of puree, clear juice, canned, chilled sections, and partially dehydrated peach products from southeastern grown peaches have indicated the more promising leads in developing products with commercial potential. The research demonstrated

that certain peach varieties are suited for the manufacture of all five type products, while some early varieties may not be suited for use in most, if any, processed products (particularly for the retail trade) because of color, flavor, soft or broken seeds, and the like. Other varieties will require special adaptation of processing procedures to make good products. Limited consumer preference tests indicate good market potential for both peach puree and chilled sections. Excellent drinks were made from the purees of frozen and canned peaches in combination with eggs, milk, ice cream, and other additives. In the canning experiments, nature of pretreatment was found to be an important factor and, in general, peaches which had been hydrocooled gave canned products superior in appearance and texture. (SU-0-0-1 (DC)).

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

None

Line Project Check List - Reporting Year July 1, 1962 to June 30, 1963

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
S1 4-	Rice Utilization Investigations-Southern Region			
S1 4-12	Investigation of the biochemical characteristics of rice as affected by and in relation to age and processing characteristics, with special emphasis on the susceptibility of rice starch to amylolytic action.*	New Orleans, La.	Yes	14-A-1
S2 1-	Cotton Utilization Investigations			
S2 1-137	Improved cleaning at the cotton card.**	New Orleans, La.	Yes	3-A-2
S2 1-140 (C)	Development of improved winter weight cotton fabrics on the woolen processing system that will successfully compete with fabrics made from synthetic fibers and blends of synthetic fibers for clothing and household use.**	Lowell, Mass.	Yes	5-E-1
S2 1-148 (C)	Investigations designed to develop reactive finishing agents for cotton that will provide improved elastic and strength properties by the introduction of, cellulose crosslinks of optimum size and structure.**	Lowell, Mass.	Yes	4-A-2
S2 1-151	A study of the etherification of cellulose using radioactively labeled etherifying reagents to contribute basic information on wash-wear cotton.**	New Orleans, La.	Yes	1-A-1
S2 1-152	A microscopical study of degradation of cotton cellulose structure by various agents.**	New Orleans, La.	No	
S2 1-153	Determination of the effect of the principal types of spotted cotton on product quality and processing efficiency to obtain optimum use of such cotton.**	New Orleans, La.	Yes	2-A-1
S2 1-154	Development of a Bale-Breaker-Blender for opening and blending cotton.	New Orleans, La.	Yes	3-A-1
S2 1-156	Development of weather- and rot-resistant cotton fabrics.	New Orleans, La.	Yes	5-A-1
S2 1-157 (C)	Design and development of acceptable cotton crepe apparel fabrics to compete with synthetic fibers in these markets.	Philadelphia, Pa.	Yes	5-F-1
S2 1-161	Crosslinking of cotton cellulose with difunctional etherifying agents using alkaline catalysts.**	New Orleans, La.	Yes	1-B-1
S2 1-162	Development of wash-wear cotton fabrics and garments with durable creases and shape holding properties.**	New Orleans, La.	Yes	4-C-1
S2 1-163	Development of optimal structures for cotton fabrics for wash-wear products	New Orleans, La.	Yes	4-B-3
S2 1-164	Development of a machine for removing short fibers from cotton.**	New Orleans, La.	Yes	3-A-3
S2 1-164 (Rev.)	Development of a prototype machine for removing short fibers from cotton.*	New Orleans, La.	Yes	3-A-3
S2 1-165	Study of the infrared absorption spectra of native, degraded, and chemically modified cotton cellulose as a means of elucidation of structural and chemical changes due to chemical treatment.**	New Orleans, La.	Yes	1-A-5
S2 1-166	Development of improved wash-wear cotton fabric by reaction with formaldehyde.**	New Orleans, La.	Yes	4-B-1
S2 1-167	Systematic exploratory investigation of chemical pretreatments as a means of producing resilient cotton fabrics having improved abrasion and tear resistance.**	New Orleans, La.	Yes	1-B-1
S2 1-168	Exploration of cellulosic crosslinks capable of being broken and reformed at will.	New Orleans, La.	Yes	1-B-1

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
S2 1-169	A study of the reaction of cotton with epoxy compounds in the presence of free radicals or disproportionating products.**	New Orleans, La.	Yes	1-B-1
S2 1-170 (C)	Investigation of the relationships between ease-of-care performance and the geometry of cotton fabrics.	Dedham, Mass.	Yes	4-B-3
S2 1-171	Application of crosslinking treatments to chemically modified cottons.**	New Orleans, La.	Yes	4-B-2
S2 1-172	Exploratory study of means of producing thermoplastic cottons.**	New Orleans, La.	Yes	1-B-1
S2 1-173 (C)	Determination of the mechanics of nep formation in cotton during textile mechanical processing	Cambridge, Mass.	Yes	1-B-4
S2 1-174	Microscopical investigation of reaction products in chemical modifications of cotton fibers.	New Orleans, La.	Yes	1-A-3
S2 1-175 (C)	Fundamental investigation of the effects of specific type finishes on soiling of and soil removal from cotton.	Washington, D.C.	Yes	5-B-1
S2 1-176	Preparation of cotton products containing radiation-induced polymers having desirable physical properties.	New Orleans, La.	Yes	1-B-2
S2 1-177	N-methylol finishes for high quality durable wash-wear cotton fabrics.**	New Orleans, La.	Yes	4-A-2
S2 1-178 (C)	Large-scale spinning evaluation of the effect of fiber properties and spinning variables on yarn properties and end breakage during spinning.	Auburn, Ala.	Yes	2-A-1
S2 1-179	Development of optimum processing procedures to minimize the detrimental effects of short fibers in cotton spinning performances and product quality.	New Orleans, La.	Yes	2-A-2
S2 1-180	Modification of cotton with fluorochemicals to impart durable water and oil repellency.	New Orleans, La.	Yes	5-B-1
S2 1-181	Improvement in the bulk resilience and cohesion of			
S2 1-181 (Rev.)	cotton batts as a means of enhancing cotton's competitive position in this market.** *	New Orleans, La.	Yes	5-E-3
S2 1-182	Evaluation of the swelling behavior of cotton fibers in various environments by observation of the untwisting of single yarns.	New Orleans, La.	Yes	1-A-2
S2 1-183 (C)	Investigation of the effects of mechanical treatments prior to, during, and following resin finishing on the ease-of-care properties of fabrics and garments.	Raleigh, N. C.	Yes	4-B-2
S2 1-184	An engineering study of the feasibility and practicality of chemical and/or resin treatment of roving by continuous processing as an intermediate step in the mechanical processing of cotton.	New Orleans, La.	Yes	5-F-1
S2 1-185	Basic investigations to characterize fiber damage in mechanical processing from opening through carding to provide information needed to develop improved textile machinery and processing methods.	New Orleans, La.	Yes	2-A-2
S2 1-186	Chemical attachment of reactive compounds to cotton cellulose by means of polyfunctional reagents	New Orleans, La.	Yes	1-B-1
S2 1-187	The development of stretchable cotton fabrics for various outdoor uses by slack mercerization.**	New Orleans, La.	Yes	5-E-2
S2 1-188	A fundamental investigation of the drying of chemically modified cotton, with emphasis on resin treated cotton, as a means of producing cotton products of superior quality.	New Orleans, La.	Yes	1-B-4
S2 1-189	Investigation of wet and dry crease recovery mechanisms in wash-wear cotton products.	New Orleans, La.	Yes	4-A-1
S2 1-190	Exploratory investigations to impart improved properties to cotton needed for specific end uses.	New Orleans, La.	Yes	5-C-1

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S2 1-191	Investigation of various finishes with respect to soiling and soil removal from cotton.	New Orleans, La.	Yes	5-B-1
S2 1-193	Development of stretchable-type cotton yarns and fabrics			
S2 1-193 (Rev.)	that will successfully compete with fabrics made from synthetic fibers and blends of synthetic fibers for clothing, household, and industrial uses.** *	New Orleans, La.	Yes	5-E-1
S2 1-194	Exploratory investigation of methods for imparting durable luster and related appearance characteristics to cotton textiles.	New Orleans, La.	Yes	5-D-1
S2 1-195	Investigation of radiochemical yields of high-energy radiation activated reactions of cotton to develop improved cotton products.*	New Orleans, La.	Yes	1-B-2
S2 1-196	Fundamental study of mechanisms of cellulose etherifications.*	New Orleans, La.	Yes	1-B-3
S2 1-197 (C)	Evaluation of stretch type cotton yarns in knit wear.** *	Raleigh, N. C.	Yes	5-E-1
S2 1-197 (C) (Rev.)				
S2 1-198	Relationship of fiber properties to fabric behavior in wash-wear treatments.*	New Orleans, La.	Yes	4-A-1
S2 1-199 (C)	Exploratory investigation of the reaction of acetylene and related compounds with cotton cellulose.*	New York, N. Y.	Yes	1-B-1
S2 1-200 (C)	Development of weather-resistant, water-repellent finishes for cotton.*	Denton, Tex.	No	
S2 1-201	An investigation of the interfiber frictional force and associated fiber properties to improve the processing of cotton products.*	New Orleans, La.	Yes	1-A-6
S2 1-202	Exploratory investigation to impart special properties to cotton through the use of lead compounds.*	New Orleans, La.	Yes	1-B-1
S2 1-203	Investigation of the effect of time and environmental conditions on the rate of wrinkle recovery of wash-wear cotton fabrics.*	New Orleans, La.	Yes	4-A-1
S2 1-204 (C)	The aerodynamic separation of lint cotton into individualized fibers to provide information needed for improving cotton textile processing equipment.*	Westbury, L.I., N. Y.	Yes	3-A-4
S2 1-205 (C)	The development of cotton knit fabric having increased bulk, warmth, and dimensional stability.*	Clemson, S. C.	No	
S2 1-206 (C)	A determination of the structural components of the cotton fiber that contribute most to tensile strength and how they can be utilized to increase tensile and recovery properties to produce cotton products having enhanced physical properties.	South Pasadena, California	No	
S2 1-207	Development of guides for the maximum utilization of cottons of varying fiber properties.*	New Orleans, La.	Yes	2-A-1
S2 1-208	Investigation of the effects of gross and fine structures of the cotton fibers on their physical behaviors.*	New Orleans, La.	Yes	1-A-3
S2 1-209	Microscopical investigations of absorption phenomena in native, mercerized, and modified cottons.*	New Orleans, La.	No	
S2 1-210	The crosslinking of various physically modified crystalline forms of cotton as a means of producing resilient cotton textiles having improved appearance and durability to wearing.*	New Orleans, La.	No	
S2 1-211	Investigation of finishing treatments to produce wash-wear cotton stretch fabrics with improved strength, drape, and hand.*	New Orleans, La.	Yes	4-C-2

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
S2 1-212	Development of test methods for stretch cotton textiles for use as a guide in producing better cotton stretch yarns and fabrics.*	New Orleans, La.	Yes	2-B-1
S2 1-213	The development by chemical and mechanical treatment of stretchable cotton yarns suitable for weaving and knitting into fabrics with enhanced qualities.*	New Orleans, La.	Yes	5-E-1, 2
S2 1-214	Separation and identification of the cleavage products of partially etherified cottons, including crosslinked cottons.*	New Orleans, La.	Yes	1-A-5
S2 1-215	Development of an improved method of feeding the cotton card to produce higher quality te tile products and thus increase the utilization of cotton.*	New Orleans, La.	Yes	3-A-2
S2 1-216	A study of reactions between epoxy compounds or their halohydrin precursors and cotton cellulose.*	New Orleans, La.	No	
S2 1-217 (C)	Effect of variation in structure on cotton fiber properties caused by environmental and genetic factors to obtain basic information important in optimum utilization of cotton.*	College Station, Texas	No	
S2 1-218	Development of improved instrumental techniques for selected elemental analysis of additively and chemically modified cottons to aid in improvement of cotton textile products.*	New Orleans, La.	No	
S2 1-219	Improved methods of etherifying cotton cellulose.*	New Orleans, La.	No	
S2 1-220	Investigation of improved infrared spectral techniques for the study of modified cottons to aid in the development of textile products for specific end uses.*	New Orleans, La.	Yes	1-A-5
S3 2-	Citrus and Other Fruit Utilization Investigations-Southern Region			
S3 2-23	Investigations to develop new and improved processed products from selected minor fruits, with emphasis on avocados, limes, and Meyer lemons.**	Weslaco, Tex. and Winter Haven, Fla.	No	
S3 2-32	Investigations on the "foam-mat" drying of concentrated citrus juices to provide new citrus products of optimum flavor and high stability.	Winter Haven, Fla.	Yes	9-C-1
S3 2-33	Investigations to develop a highly colored frozen grapefruit concentrate and grapefruit drinks from colored grapefruit by pulp fortification.**	Weslaco, Tex.	Yes	9-B-2
S3 2-34	Investigation of the origin of carotenoid precursors and the biochemical mechanism of their conversion to carotenoids in grapefruit as a basis for improvement of processing characteristics of and products from colored grapefruit.**	Weslaco, Tex.	Yes	9-A-4
S3 2-34 (Rev.)	Investigation of the biochemical mechanism of the conversion of precursors to carotenoids in grapefruit as a basis for improvement of processing characteristics of and products from colored grapefruit.*	Weslaco, Tex.	Yes	9-A-4
S3 2-35	Investigations on preservation of chilled citrus products to prevent spoilage and permit delivery of improved products.**	Winter Haven, Fla.	Yes	9-B-1
S3 2-36	Investigations on composition of essential citrus oil as related to flavor of juices, concentrates, powdered juice and other products with special emphasis on essential orange oil.	Winter Haven, Fla.	Yes	9-A-1

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** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S3 2-37	Investigations of the neutral fraction of orange peel extract for the isolation of bitter principles.	Winter Haven, Fla.	Yes	9-A-2
S3 2-38	Investigation of the chemical nature and physical state of components of cloud of orange juice, with a view to better understanding and control of factors affecting stability of frozen concentrated orange juice.	Winter Haven, Fla.	Yes	9-A-3
S3 2-39 (C)	Investigation of the effect of maturity of grapefruit on total flavonoids, naringin, and poncirin; and on the chemistry, and nature of naringin and naringin-derived compounds to provide a scientific basis for the control of bitterness in processed grapefruit products.*	Norman, Okla.	Yes	9-A-2
S3 2-40	Utilization of natural and debittered grapefruit juice and puree as bases for the development of improved fruit juice blends, drinks, and concentrates.*	Weslaco, Tex.	Yes	9-B-2
S3 2-41	Investigations on the foam-mat drying of concentrated grapefruit juices to provide a new grapefruit product of optimum flavor and high stability.*	Winter Haven, Fla.	Yes	9-C-1
S3 2-42	Investigations of the identities, quantities and chemistry of components in Florida grapefruit responsible for excessive bitterness and harshness in processed products.*	Winter Haven, Fla.	No	
S3 5-	Sweetpotatoes, Cucumbers, and Other Vegetable Utilization Investigations-Southern Region			
S3 5-16	Investigations to develop new and improved processed products from vegetables with emphasis on cucumbers, snapbeans, southern peas, tomatoes and spinach.**	Raleigh, N.C. and Weslaco, Tex.	Yes	10-C-2 10-B-2
S3 5-17	Investigations to develop improved processed products from celery with emphasis on flavor components.**	Winter Haven, Fla.	Yes	10-A-2
S3 5-19	Development of a practical pilot plant process for precooked dehydrated sweetpotato flake products, with improved product quality and processing efficiency to extend the utilization of sweetpotatoes.	New Orleans, La.	Yes	10-B-1 10-C-1
S3 5-20	Investigation of methods for the controlled fermentation of cucumbers with emphasis on the application of pure culture techniques to reduce processing costs and improve product characteristics.	Raleigh, N.C.	Yes	10-C-2
S3 5-21	Investigation of the flavor and aroma components in natural and pure culture fermented cucumber pickle products.*	Raleigh, N.C.	Yes	10-A-1
S3 5-22	Investigation to develop new and improved processed products from southern grown vegetables other than sweetpotatoes and celery, including cooperative studies with federal, state, and industry agencies.*	Raleigh, N.C. and Weslaco, Tex.	Yes	10-C-2
S3 5-23	Application of new basic information on the chemical constituents of celery stalk (petiole) to the development of processed products of improved flavor and convenience.*	Winter Haven, Fla.	Yes	10-A-2 10-B-2
S4 1-	Cottonseed, Peanut and Other Oilseed Investigations-Southern Region			
S4 1-88	Investigations of solubilities of long-chain fatty acids and their derivatives important to research and industrial utilization of fatty acids from vegetable oils of Southern Region.	New Orleans, La.	No	

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** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S4 1-89 (C)	Polymerization of vegetable oil, pine gum, and sugarcane derivatives and evaluation of properties of the polymers for use as elastomers, plastics, thickening agents, and protective coatings.	Tucson, Ariz.	Yes	11-B-3
S4 1-90	New polyester products from cottonseed oil for use as edible and inedible coating materials, waxes, resins, plasticizers, and lubricants.	New Orleans, La.	Yes	6-B-1
S4 1-91	Preparation of new and improved confectionery fats and oils from cottonseed and peanut oils.**	New Orleans, La.	Yes	6-B-1
S4 1-92	Pilot-plant scale development of a process for improving cottonseed oil color based on highly active adsorbents.	New Orleans, La.	Yes	6-B-2
S4 1-93	Chemical modification of tung oil and its fatty acids to produce materials having utility as protective coatings, agricultural chemicals, surfactants, or plasticizers.	New Orleans, La.	Yes	8-B-2
S4 1-94	Pilot plant investigations of cottonseed processing using hexane-acetone-water solvent mixtures to improve meal and oil quality.**	New Orleans, La.	Yes	6-C-2
S4 1-95	Development of new information basic to the production of cottonseed meals that can be fed to swine and poultry without restriction.**	New Orleans, La.	Yes	6-C-1, 2
S4 1-96	Investigations on the improvement of the color and quality of off-colored cottonseed oils.**	New Orleans, La.	Yes	6-A-2, 3
S4 1-97	Investigation of physiologically active constituents in cottonseed meal that affect the utilization of the meal as a protein supplement in nonruminant feeding.**	New Orleans, La.	Yes	6-C-1
S4 1-98	Development of exterior intumescing fire-retardant surface coatings from tung oil alkyds.**	New Orleans, La.	Yes	8-B-1
S4 1-99	Investigation of long-chain fatty amides and derivatives potentially useful as plasticizers, polyurethane foams, and other industrial uses.	New Orleans, La.	Yes	6-D-1
S4 1-100	Investigations of the constituents and their modifications by processing that influence nutritive properties and consumer acceptance of processed peanut products.	New Orleans, La.	Yes	7-A-2
S4 1-101	Engineering studies to develop a commercial process for preparing cocoa butter-like fat from cottonseed oils.	New Orleans, La.	Yes	6-B-2
S4 1-102	Development of hydrogenation techniques which will produce the least possible isomerization in edible fat products prepared from cottonseed oil and peanut oil.**	New Orleans, La.	No	
S4 1-102 (Rev.)	Development of hydrogenation techniques for cottonseed oil which will reduce cyclopropenoids with the least possible isomerization of the other unsaturated fatty acid groups.*	New Orleans, La.	No	
S4 1-103 (C)	Investigations of gossypol esters and of mild oxidation products of gossypol and gossypol derivatives to develop information needed to aid the production of cottonseed meals and oils of the highest quality.	Knoxville, Tenn.	Yes	6-A-2
S4 1-104 (C)	Investigation of the chemical and physical properties of cyclopropene fatty acids in cottonseed.	Urbana, Ill.	Yes	6-A-2
S4 1-105	Investigations on the cyclopropene acid constituents in cottonseed and cottonseed products.*	New Orleans, La.	Yes	6-A-2, 3 6-B-2

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S4 1-106 (C)	Investigation of the flavor and aroma components in processed peanut products.*	New York, N. Y.	Yes	7-A-2
S4 1-107 (C)	Chemical investigations of cyclopropenoids to develop means of eliminating or physiologically inactivating the cyclopropenoids found in cottonseed products.*	Urbana, Ill.	Yes	6-B-2
S4 1-109	Investigations of the proteins and nonglyceride lipid-soluble constituents of peanuts and processed peanut products to expand their utilization.*	New Orleans, La.	Yes	7-A-2
S4 1-110	Investigations to isolate and identify the factors in cottonseed meal that cause mortalities among swine to develop information for producing cottonseed meals that can be used without restriction in feeding to any nonruminants.*	New Orleans, La.	Yes	6-C-1
S4 1-111	Pilot-plant development of a cottonseed extraction process using hexane-acetone-water solvent mixtures to a stage suitable for commercial evaluation.*	New Orleans, La.	Yes	6-C-2
S4 1-112 (C)	Investigations of chemical transformations of fat and terpene olefinic compounds by hydroboration and suitable subsequent reactions to produce useful products.*	Lafayette, Ind.	No	
S5 1-	Sugars and Sirups Investigations-Southern Region			
S5 1-70	Pilot-plant development of new and improved methods of clarification and processing to increase the recovery and improve the quality of raw cane sugar.**	New Orleans, La. and Baton Rouge, La.	Yes	12-B-1
S5 1-71	Investigation of the composition of sugarcane in relation to processing efficiency.	New Orleans, La. and Houma, La.	Yes	12-A-1
S5 2-	Naval Stores Investigations-Southern Region			
S5 2-36	The isolation and characterization of some of the major unidentified components in pine gum, rosin and some of their derivatives.**	Olustee, Fla.	Yes	11-A-1
S5 2-37	Photochemical addition of suitable reagents to resin acids of pine gum origin to produce new chemicals of potential utility in the fields of surface active agents, textiles, paper and plastics.**	Olustee, Fla.	Yes	11-B-2
S5 2-38	Preparation of chemical intermediates from pine gum products for use in the preparation of new synthetic polymers, plastics and resins to expand the utilization of turpentine and rosin.	Olustee, Fla.	Yes	11-B-1
S5 2-39	Development of a method for the determination of rosin and rosin derivatives in protective coatings.	Olustee, Fla.	Yes	11-B-3
S5 2-40	Production of reactive chemical intermediates from turpentine by reaction with selected low molecular weight reagents.	Olustee, Fla.	Yes	11-B-1
S5 2-41	Development of process for isolation of levopimaric acid from pine gum.	Olustee, Fla.	Yes	11-C-1
S5 2-42	Polyester resins from pine gum derivatives.	Olustee, Fla.	Yes	11-B-3
S5 2-43	The preparation of polyfunctional compounds from rosin, resin acids, and resin acid derivatives through their reaction with formaldehyde.	Olustee, Fla.	Yes	11-B-3
S5 2-44	Hypochlorite modification of rosin and resin acids for use as chemical intermediate for preparation of new industrial resins, surface coatings, plastics, rosin soap emulsifiers and similar materials.*	Olustee, Fla.	Yes	11-B-3

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** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S5 2-45 (C)	Application of the oxo and related reactions to terpenes and resin acids to produce alcohols, aldehydes, and/or acids and characterization of the products thus obtained.*	Cincinnati, Ohio	No	
S5 2-46 (C)	Synthesis of terpene alcohols and glycols for use in the production of new and useful terpene derived polymers.*	Ithaca, N. Y.	No	
S5 2-47	The utilization of photosensitized oxidized pine gum and components in the fields of plastics and rubber.*	Olustee, Fla.	Yes	11-B-2
S5 5-	New and Replacement Crops Utilization Investigations-Southern Region			
S5 5-45	Investigation on chemical modification of oils from potential new oilseed crops such as Limnanthes and Cuphea to produce materials having utility in plastics, plasticizers, synthetic rubbers, protective coatings, and other industrial products.	New Orleans, La.	Yes	13-A-1
SG-0-1	Development of emulsifiable oils and fats for use in intravenous alimentation, for the Office of the Surgeon General-Southern Region.***	New Orleans, La.	Yes	6-B-1
SU-0-0-1 (DC)	Development of new and improved processed peach products, with special emphasis on the suitability of existing commercial varieties in the Southeast for the production of fresh peach concentrates.***	Griffin, Ga.	Yes	15-
SU P 1	Seed Protein Pioneering Research Laboratory.***	New Orleans, La.	Yes	7-A-1
SU P 2	Plant Fibers Pioneering Research Laboratory.***	New Orleans, La.	Yes	1-A-5
UR-A7-(40)-3	A study of the relationship of substituent fatty acid groups on the physical properties of diacid triglycerides of palmitic and stearic acids as a means of increasing the utilization of cottonseed oil for food and industrial purposes.	Bombay, India	Yes	6-A-3
UR-A7-(20)-4	Investigation of the photochemical degradation of cotton to derive information which would enhance the utilization of cotton.	Bombay, India	Yes	1-A-4
UR-A7-(40)-12	Effect of heat on tung oil and derivatives of tung oil and characterization and identification of compounds resulting from heat treatments, to extend the utilization of tung oil.	Poona, India	No	
UR-A7-(20)-19	A study of the relation between fine structure and mechanical properties of cotton fibers by swelling and stretching treatments, as a means of improving the properties, and thereby increasing the utilization of cotton.*	Ahmedabad, India	No	
UR-A7-(40)-26	Studies of the addition of carbenes to unsaturated fatty materials derived from cottonseed oil, to provide possible new outlets for the utilization of cottonseed oil.*	Bangalore, India	No	
UR-A7-(40)-28	Investigation of the synthesis and properties of new-type glycol mono alkyl ethers for the control of water evaporation, to extend the industrial utilization of cottonseed oil.*	Poona, India	No	

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*** There are no line projects under this work project.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
UR-A7-(20)-30	Investigation of new solvents for molecular weight determination of cellulose, to obtain basic information needed to improve cotton products and thereby enhance the utilization of cotton.	Bombay, India	No	
UR-A7-(20)-32	Investigation of the microbial decomposition of cellulose with special reference to the effect of Indian bacterial organisms on cotton and cotton fabrics to provide basic information for the improvement of cotton products.*	Bombay, India	No	
UR-A10-(20)-4	A fundamental study of oxidation of cotton by hypochlorite, hypobromite, hypoiodite, and other oxidizing agents to obtain data on kinetics of oxidation and changes in physical and chemical properties, as a contribution to improved uses for cotton.**	Jerusalem, Israel	Yes	1-B-3
UR-A10-(20)-5	Fundamental investigation of crimp in cotton fibers and its relationship to other fiber properties, as well as its effect on processing performance and product quality.	Jerusalem, Israel	Yes	1-A-6
UR-A10-(40)-34	Investigation of π-complexed organometallic compounds derived from polyunsaturated fatty acids, to obtain fundamental information needed in expanding the utilization of cottonseed oil.*	Haifa, Israel	No	
UR-A11-(50)-7	Isolation and identification of the nucleic acid derivatives of cane molasses, in order to obtain information applicable to expanding the utilization of molasses industrially and in feeds.*	Kyoto, Japan	No	
UR-E9-(00)-29	Investigation of preparation and properties of alkyl aryl ketones and their derivatives from vegetable oils and animal fats to provide information of potential value in increasing utilization of these commodities in such industrial products as surface-active agents, lubricants, plasticizers and fungicides.**	Paris, France	Yes	6-D-1
UR-E9-(20)-61	A fundamental study of the relation of crystallinity to accessibility in native and modified cotton, to obtain information on the supermolecular structure of cotton that is needed in the development of improved cotton products.	Paris, France	Yes	1-A-5
UR-E10-(20)-2	Development of an apparatus for counting neps in cotton card web as an aid toward increasing the quality of cotton products.	Reutlingen-Stuttgart, West Germany	Yes	2-B-1
UR-E15-(40)-33	Investigations on the physical and physicochemical properties of cottonseed proteins, to obtain basic information needed for the increased utilization of cottonseed.*	Rome, Italy	No	
UR-E19-(20)-4	A fundamental study of the role of the structural elements of the cotton fiber in response to stress in deformation and recovery, to obtain information needed in the development of improved cotton products.	Delft, Holland	Yes	1-A-3
UR-E25-(20)-1	Development of methods and equipment for determining irregularity of transparency of card web and for counting of neps, by means of electronic devices, as aids to improving product quality in cotton textile operations.	Barcelona, Spain	Yes	2-B-1
UR-E25-(20)-2	Determination of relationship between the cohesion of cotton fibers and other physical properties of fibers, rovings, and yarns, as a step in improving product quality and processing efficiency.	Barcelona, Spain	Yes	2-A-1

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** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
UR-E25-(20)-13	Determination of effect of drafting forces in high-draft systems on uniformity and strength of cotton yarns as a step in improving product quality and processing efficiency.	Barcelona, Spain	Yes	2-A-2
UR-E26-(20)-2	Fundamental investigation of setting reactions for cotton fabrics and garments, to develop information basic to the improvement of cotton products, thereby increasing the utilization of cotton.	Gothenburg, Sweden	Yes	1-A-3
UR-E29-(20)-4	Fundamental investigation of the causes of warp breakage in the weaving of cotton yarns, as a basis for improving quality and reducing costs of production.	Didsbury, Manchester, England	Yes	2-A-1
UR-E29-(20)-6	Fundamental study of the microbiological breakdown of natural cotton fiber, as a contribution to the better preservation of cotton products.	Didsbury, Manchester, England	Yes	1-A-4
UR-E29-(20)-9	Fundamental study of the pyrolysis of cotton cellulose to provide information needed for improvement of flame-resistant treatments for cotton.	Didsbury, Manchester, England	Yes	1-B-3
UR-E29-(40)-26	Studies on the fatty acid and glyceride composition of cottonseed oil and the crystallizing behavior of some of the major components, to obtain fundamental information that will contribute to the development of improved edible products and hence to expanded utilization of cottonseed oil.	Leatherhead, Surrey, England	Yes	6-A-3
UR-E29-(20)-35	Fundamental investigation of preparation and properties of esters, anhydrides, hydrazides, pseudohalides, fluorides, and related compounds of the phosphonitrilic chlorides for use in preparing new products to increase the utilization of cotton.	London, England	Yes	1-B-1
UR-E29-(20)-55	A fundamental study of the preparation and properties of phosphazene (phosphonitrilic) and phosphoryl chloride derivatives having potential for reaction with cotton cellulose, to obtain information needed in the development of new useful products from cotton, thus increasing its utilization.	London, England	No	
UR-S9-(40)-2	Preparation, characterization, and evaluation of derivatives of gossypol from cottonseed for use as biologically active materials, ultraviolet absorbers and other valuable products.	Montevideo, Uruguay	Yes	6-D-1